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BUREAU OF MINES
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INFORMATION CIRCULAR

GERMAN LOW-TEMPERATURE COAL-TAR INDUSTRY

BY

E. O. RHODES

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FOREWORD

This report is one of a series written by members of the Solid Fuels Mission to Germany describing wartime developments in the mining, preparation, and utilization of coal. This mission was organized early in 1945 under the auspices of the Technical Industrial Intelligence Committee, which cooperated with a counterpart British Committee. In some instances, engineers from other allied countries cooperated. The Solid Fuels Mission operated under the direction of a steering committee headed by Dr. C. J. Potter, Deputy Solid Fuels Administrator. The personnel of the mission consisted of H. F. Yancey, in charge, J. W. Buch, Thomas Fraser, L. L. Newman, and L. D. Schmidt of the Bureau of Mines; H. H. Lowry, Director of the Coal Research Laboratory, Carnegie Institute of Technology; H. J. Rose, Vice-President and Director of Research, Bituminous Coal Research, Inc.; and F. H. Reed, Chief Chemist, Illinois Geological Survey.

E. O. Rhodes, Manager, Development Section, Tar Products Division, Koppers Co., Inc., served on the mission for the Chemical Subcommittee of T.I.I.C. in Europe. His assignment was the investigation of the coal-tar industry. In this connection he prepared this report on the German Low-Temperature Tar Industry as a companion to his previous report on the German High-Temperature Tar Industry (Bureau of Mines Information Circular 7409). Because of Mr. Rhodes' extensive experience in these fields and the importance of the subject material to the coal producers and those utilizing coal-tar products, both reports are issued by the Bureau of Mines.

Over fifty reports were written by the members of the Solid Fuels Mission. These, together with microfilms of supporting data obtained in the course of the investigations, are on file at the Foreign Documents Group, Technical Reports Section, Bureau of Mines, Pittsburgh, Pa., where they are available for examination. Copies of reports available in printed form may be ordered from the Department of Commerce, Office of Technical Services, Washington 25, D. C. Check or money order accompanying order for printed reports should be made payable to the Treasurer of the United States.

A Bibliography of Scientific and Industrial Reports, covering all materials and subjects and giving instructions for obtaining the desired items, has been published weekly by the Office of Technical Services. Effective July 1, 1948, the Bibliography of Scientific and Industrial Reports will be prepared monthly instead of weekly and on a more selective basis. The July issue, which will be the first monthly issue, will be available about July 25, 1948. Succeeding monthly issues will appear about the 25th of each month thereafter through June 1949. Annual subscriptions and orders for individual copies starting with the July 1948 issue should be addressed to the Department of Commerce, Office of Technical Services, Washington 25, D. C., and be accompanied by check or money order made payable to the Treasurer of the United States. Annual subscriptions for monthly issues of the Bibliography of Scientific and Industrial Reports will be $10. Individual issues will cost $1 per copy. Foreign subscriptions will be $14 per year and $1.50 per copy.

All orders for reports in microfilm or photostatic type of reproductions listed in the Bibliography of Scientific and Industrial Reports should

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be addressed to the Library of Congress Photoduplication Service, Publication Board Project, Washington 25, D. C. Check or money order accompanying orders for microfilm or photostat reports should be made payable to the Librarian of Congress.

The reports of the Solid Fuels Mission relate to the following general subjects: Mining methods in the Ruhr district of Germany, with special reference to mechanical cutting and loading equipment, pneumatic packing, and Diesel mine locomotives; activities of industrial research laboratories supported by the German coal and coke industry; coal preparation in western Europe; low-temperature carbonization processes; metallurgical coke from weakly coking coals; low-ash electrode carbon from coal; new developments in the production of metallurgical coke and the utilization of gas and by-products; briquetting brown coal and bituminous coal without the use of binders; recent engineering developments in gas turbines and steam generators, with special reference to use of coal; German coal-fired heating and cooking stoves; and the commercial extraction of coal with solvents for production of synthetic liquid fuels or chemical products.

Numerous reports on the activities of German synthetic liquid-fuels plants and the plants for the production of synthesis gas from solid fuels are available in the same office as the solid fuels reports.

It is planned to publish in this series of Bureau of Mines Information Circulars the more important reports or combination of reports prepared by the members of the Solid Fuels Mission. A similar series on synthetic liquid-fuel activities is being prepared.

A. C. FIELDNER, Chief,
Fuels and Explosives Division

RALPH L. BROWN, Chief,
Coal Branch.

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UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

GERMAN LOW-TEMPERATURE COAL-TAR INDUSTRY[1]

By E. C. Rhodes[2]

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1/ Work on manuscript completed June 25, 1948. The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7490." Originally issued as FIAT Final Report 703 Revised, PB 91,998, and FIAT Microfilm Reel C-98, PB L 13,093, or TOM Microfilm Reel 152.

2/ Manager, Development Section, Tar Products Division, Koppers Co., Inc.

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INTRODUCTION

Part I of the following report summarizes information concerning low-temperature coal-carbonization and the coal-tar industry of Germany that was collected in Germany between September 15, 1945, and January 15, 1946. Part II contains copies of brief reports that were written following each plant visit or interview. Part III lists all of the documents collected in the course of the investigation. They are recorded in FIAT Microfilm Reel C-98 or TOM Microfilm Reel 152 (PB L 13,093). The report number PB L 13,093 was assigned by the Publications Board, United States Department of Commerce, to catalogue this Field Intelligence Agency Technical (FIAT)

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The newer processes in commercial use in Germany for the low-temperature carbonization of coal were known in the United States before the war. For this reason and because they had already been reported by other investigators, no special effort was made to obtain detailed information about them. Instead, the industry as a whole was investigated, and inquiries were made to see if other processes for carbonizing coal at low temperatures or new methods for the processing and use of low-temperature tar were being developed during the war years.

The investigation disclosed the fact that Germany's increased demands for liquid fuels between 1933 and 1945 brought about the development of a low-temperature, coal-carbonization industry, which by 1943 was producing more coal tar than the long-established German high-temperature, coal-carbonization industry. Additional plants were under construction when the war ended.

New low-temperature plants constructed between 1933 and the beginning of the war were mostly of the Lurgi-Spülgas type. The type of coal carbonized was principally brown coal. Two plants of the Krupp-Lurgi type to carbonize coking bituminous coal also were installed during this period. The capacity of one of these plants was increased during the war.

Plants that were under construction when the war ended were all intended to carbonize bituminous coal. Metal retorts with movable walls were being installed at two locations by Brennstoff Technik. Two plants using continuous, vertical, ceramic ovens were being constructed by Heinrich Koppers and Didier Werke, respectively.

In connection with low-temperature tar, it was found that approximately 76 percent was hydrogenated to make motor fuels, about 1 percent was used directly in fuel oils, and about 23 percent was processed to obtain fuel oils and paraffin. Most of the processing was carried out in old distilling and dewaxing plants, but one new plant was installed during the war at Espenhain, which employed improved distilling methods and an improved solvent-extraction process developed by Edeleanu Gesellschaft.

Waste-water disposal from low-temperature carbonization plants and hydrogenation plants became troublesome, and methods for removing phenols from the water were investigated. A new method, called the phenosolvan process, was developed by Lurgi and I. G. Farbenindustrie.

The recovery of crude-tar acids from waste water led to the installation of equipment, particularly at Leuna, by I. G. Farbenindustrie for producing phenol, cresols, xylenols, and pyrocatechol. A part of the phenol was converted

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to cyclohexanol, cyclohexanone, caprolactam, adipic acid, and Igamids. The pyrocatechol was recovered in semicommercial quantities. Plans were being formulated for larger-scale production when the war ended.

ACKNOWLEDGMENTS

The assistance of various members of the Central Experiment Station, United States Bureau of Mines, and of the Koppers Co., Inc., in the preparation of this report for issuance as an Information Circular by the Bureau of Mines is gratefully acknowledged. Dr. R. E. Brewer and W. L. Ruckes, Bureau of Mines, and Dr. John O'Brochta and Miss Edith Sullivan, Koppers Co., Inc., assisted the author in editing the report. The illustrations were prepared by members of the Graphic Services Section, Bureau of Mines, under the supervision of Louis F. Perry. The manuscript was typed by secretaries of the Bureau of Mines.

PURPOSE AND SCOPE OF THE INVESTIGATION

The primary purpose of the investigation covered by this report was to determine to what extent the production and use of low-temperature tar in Germany had advanced during the 10-year period prior to the close of the last war. It supplemented an investigation of the high-temperature coal-tar industry of Germany that was reported separately in FIAT Final Report 729 (PB 33,270), issued February 6, 1946, and later in Bureau of Mines Information Circular 7409, issued in September 1947. Both investigations were conducted during the period between September 15, 1945, and January 15, 1946.

The production and processing of high- and low-temperature coal tars in Germany in the years immediately preceding and during the last world war was carried out in two separate industries. Low-temperature tar production and processing plants were not associated with the plants producing or processing high-temperature tar. Furthermore, low-temperature tars came almost entirely from brown coal, whereas bituminous coal was the source of all of the high-temperature tar. The low-temperature tar plants and the high-temperature tar plants had one point in common. Both were so operated as to produce maximum quantities of oils suitable for use directly as fuel oils or as raw materials for the manufacture of motor fuels by hydrogenation.

This report, dealing only with the low-temperature coal-tar industry of Germany, will discuss briefly the history of low-temperature carbonization in Germany, the carbonization processes by which low-temperature tar was produced, the quantities of tar made and their uses, processing methods, the recovery of phenols from low-temperature carbonization waste waters and light oils, and the manufacture of chemicals from these phenols.

SOURCES OF INFORMATION

The information contained in this report was obtained by interviewing the individuals and members of the organizations listed in the following table:

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Company or individual	Location
Lurgi Gesellschaft für Wärmetechnik m.b.H. ..	Frankfurt a.M.
Edeleanu Gesellschaft m.b.H.	Berlin
Fried. Krupp Aktiengesellschaft	Essen
Krupp Treibstoffwerke G.m.b.H.	Wanne-Eickel
Braunschweigische Kohlen Bergwerke	Helmstedt
I. G. Farbenindustrie	Merseburg-Leuna
Braunkohle-Benzin A. G.	Berlin
Brennstoff Technik	Essen
Dr. Lameck	Essen
Firma Carl Still	Recklinghausen
Gesellschaft für Teerverwertung m.b.H.	Duisburg-Meiderich
Heinrich Koppers G.m.b.H.	Essen
Julius Pintsch Kommanditgesellschaft	Berlin
Dr. Reerink	Essen
Dr. C. Otto and Company G.m.b.H.	Dalhausen
Deutsche Erdöl A. G.	Berlin
Didier Werke A. G. (Dr. Thau)	Berlin

Brief reports covering the interviews with each of the companies and individuals listed above are presented in Part II.

The only low-temperature carbonization plants inspected were the Krupp-Lurgi installation of Krupp Treibstoffwerke at Wanne-Eickel and the Lurgi-Spülgas plant of Braunschweigische Kohlen Bergwerke at Helmstedt. Permission could not be obtained to visit any of the other German low-temperature carbonization plants, with the possible exception of the Krupp-Lurgi plant at Velsen. No attempt was made to inspect it, because time did not permit and because it was reported to be like the one at Wanne-Eickel. The two plants that were visited were typical of plants that produced approximately 93 percent of Germany's low-temperature tar.

The Krupp-Lurgi process (employed at Wanne-Eickel and Velsen) was the only one actually used during the war for carbonizing bituminous coking coal at low temperatures. The plant visited at Wanne-Eickel was typical of the two plants of that type. The Lurgi-Spülgas plant visited at Helmstedt was typical of all plants of that type used throughout Germany for carbonizing noncaking bituminous coal, hard brown coal, and brown-coal briquets.

Other types of low-temperature carbonization plants were under construction when the war ended, particularly for the carbonizing of noncaking bituminous coal. To obtain first-hand information about those plants and about low-temperature plants in general, the following engineering firms were interviewed: Lurgi, Heinrich Koppers, Brennstoff Technik, Didier Werke (Dr. Thau), and Dr. C. Otto and Company. Each of these firms had had extensive experience in this field and was able to furnish pertinent information.

Information about the processing of low-temperature tar was received from the Carl Still organization, Deutsche Erdöl A. G., Edeleanu Gesellschaft, Gesellschaft für Teerverwertung, Dr. Reerink, and Dr. Lameck. The information from Firma Carl Still was particularly pertinent, because that company built

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the only plant installed in Germany during the war for processing low-temperature coal tar. The plant was erected at Espenhain for the processing of tar from brown coal.

During the course of the investigation it was found that one of the more important and interesting phases of the low-temperature carbonization industry was the extraction and processing of phenols contained in the water leaving brown-coal hydrogenation and low-temperature carbonization plants. Several methods for extracting the phenols were used, the newest of which was the phenolsolvan process developed by Lurgi and I. G. Farbenindustrie. Information about this process and about the other methods used in Germany was collected from Lurgi, Koppers, Still, and I. G. Farbenindustrie at Leuna. Previously, information concerning the recovery of phenols from coke-plant liquors had been received from Emschergenossenschaft, Koppers, and Carl Still.

The largest plant in Germany for processing phenols extracted from industrial waters is that of I. G. Farbenindustrie at Merseburg-Leuna. From phenol recovered from waste waters from brown-coal hydrogenation and carbonization plants, and from oils produced by the hydrogenation of brown coal, the following chemicals are made at Leuna: cyclohexanol, cyclohexanone, oxime, caprolactam, and adipic acid. Descriptions of the methods used for the production of these chemicals were obtained at Leuna.

In connection with the processing of tar acids from hydrogenation and low-temperature carbonization plants, an interesting development was the recovery of pyrocatechol or brenzcatechin. Phenols extracted from the water produced at Most (Brüx) and processed at Leuna were particularly rich in this chemical, and a large amount of research was conducted on its recovery and use. Pertinent documents were obtained from Dr. F. A. Croy of Mannheim, from Dr. Günther Spengler of Munich, and from the low-temperature carbonization plant at Most (Brüx). The information from Dr. Spengler and documents concerning operations at the Lurgi-Spülgas plant at Most (Brüx) were collected by a FIAT team from the Liquid Fuels Group and contributed by it to this investigation.

Incidental to the furnishing of information about low-temperature tar, the Julius Pintsch organization supplied some information about the Pintsch-Hillebrand generator developed by that firm.

In connection with low-temperature carbonization in Germany, the Büttner drier has some important applications, so documents on that subject (collected by a team from the Solid Fuels Group) were added to the documents collected during this survey.

The documents received from all the sources mentioned above were so numerous and voluminous that they cannot be discussed in detail or included in their entirety in this report. On this account they were microfilmed, and an index of the microfilmed documents (FIAT Reel C-98 or TOM Reel 152, PB L 13,093) is attached at Part III of the present report.

A summary of the more pertinent information obtained from the various interviews and documents, with emphasis on those phases of the subject that

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have to do with recent developments, follows in Part I of this report. Throughout this summary reference has been made, by number, to the frames in FIAT Microfilm Reel C-98 or TOM Microfilm Reel 152 (PB L 13,093) that furnish more detailed information concerning the various points mentioned in this report.

PART I. - PRODUCTION, PROCESSING, AND USE OF GERMAN LOW-TEMPERATURE TAR

History of Low-Temperature Carbonization in Germany

The history of low-temperature carbonization in Germany may be divided roughly into four periods, as follows: First period, 1850-1918; second period, 1818-1933; third period, 1933-1941; and fourth period, 1941-1945.

First Period, 1850-1918

Low-temperature carbonization was first applied to the coking of brown coal in Germany about 95 years ago. Horizontal metal retorts were used for the purpose. In 1872, a vertical oven of circular cross section was developed by Dr. Rolle. It superseded the horizontal retorts used prior to that time and was adopted almost exclusively during the remainder of this period.

Second Period, 1918-1933

The low-temperature carbonization of bituminous coal began in Germany in 1918 owing to an acute shortage of mineral oils. Three different continuous processes, each involving the use of rotating inclined retorts, were developed. The semicoke produced by these processes was of poor quality, and the tar was contaminated with coal dust. However, because oil was needed at any cost, these uneconomical processes were operated until fuel and oil markets returned to normal. They were then given up almost completely.

During this period the throughput of the Rolle ovens, developed in the preceding period, was increased by carbonizing brown-coal briquets instead of raw brown coal. Also, in 1924 Geissen introduced an oven with a revolving, vertical, corrugated, cast-iron cylinder. Predried, screened, brown coal was carbonized in this equipment. Several plants of this type were still operating in Germany at the end of World War II.

Third Period, 1933-1941

During this period the continued need for increased quantities of fuel oils of all kinds renewed interest in low-temperature carbonization. An improved retort was introduced by Geissen in 1935 and built by Borsig under the name Borsig-Geissen oven. Only a few plants of this type were erected. Both Lurgi and Kollergas developed processes for carbonizing noncaking bituminous coal and brown-coal briquets, in which carbonization was carried out by means of hot gases circulated through a bed of the coal to be carbonized.

Also, during this period the Krupp and Lurgi organizations jointly developed the Krupp-Lurgi process, in which caking bituminous coal is heated

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indirectly and intermittently in slotted steel retorts. Brennstoff-Technik also developed a metal oven for carbonizing caking bituminous coal. It was equipped with movable walls to facilitate removal of the coke by gravity at the end of the coking period.

Fourth Period, 1941-1945

The ever-increasing need for fuel oils during the war caused other methods for carbonizing coal at low temperatures to be considered, and three firms experienced in the construction of ceramic ovens collaborated in the development of vertical ceramic ovens suitable for the low-temperature coking of noncaking bituminous coal. Large installations were under construction by Heinrich Koppers G.m.b.H. and Didier Werke A. G. in Upper Silesia when the war ended.

More detailed information concerning the history and development of low-temperature carbonization in Germany is contained in a statement written by Dr. Adolf Thau for use in the preparation of this report. The statement is entitled "The State of Low-Temperature Carbonization in Germany Before the Conclusion of the War" (frames 1-30; 31-301). Additional information is given in Dr. Thau's book, "Kohlenschwelung" (frames 202-418), and in Amt für Technik der N.S.D.A.P. "Bericht über die Tagung 'Heimische Treibstoffe,' Nürnberg, October 1936" (frames 425-482).

Low-Temperature Carbonization Plants in Operation at End of World War II

The types of low-temperature carbonizing plants used in Germany during World War II, their locations, and the quantities of tar produced by each in 1944 are summarized in table 1, the information for which was furnished by the Lurgi Geselschaft für Wärmetechnik m.b.H.

No attempt will be made in this report to describe in detail the various German low-temperature carbonization processes listed above. Most of them have been described in articles published before the war. Following are brief statements concerning each of them and references to the frames in FIAT Microfilm Reel C-98 or TOM Microfilm Reel 152 (PB L 13,093), where more detailed information is given.

Lurgi-Spülgas Process: Frames 3-5; 93-97; 231-234; 264-266; 385-389; 525-534; 767-773; 774-785; 971-972.

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TABLE 1. - Low-temperature carbonization plants operating in Germany in 1943-1944

Group No.	Kind of coal, company, and process	Tar production, tons (metric) per year[1/]
I	Coking bituminous coal	
	Krupp-Lurgi process	
	Treibstoffwerke, Wanne-Eickel	20,000
	Saargruben Aktiengesellschaft, Velsen	8,000
		28,000
II	Caking, noncoking, bituminous coal	
	Lurgi-Spülgas process	
	I. G. Farbenindustrie, Oswiecim (Auschwitz)	60,000
	O.H.W. Blechhammer	140,000
	Karsten-Zentrum Grube, Beuthen	5,000
		205,000
III	Soft brown coal	
	Lurgi-Spülgas process	
	Braunschweigische Kohlenwerke, Offleben	120,000
	Riebecksche Montanwerke, Nachterstedt	50,000
	Riebecksche Montanwerke, Deuben	80,000
	Deutsche Erdöl, Regis	130,000
	Braunkohlenwerke Salzdetfurth, Deutzen	90,000
	Akt. Sächsische Werke, Espenhain	350,000
	Akt. Sächsische Werke, Böhlen	280,000
	Akt. Sächsische Werke, Hirschfelde	70,000
	Anhaltische Kohlenwerke, Profen	40,000
		1,210,000
	Borsig-Geissen process	
	Leipziger Braunkohlenwerke, Kulkwitz	40,000
	Rolle, old Geissen, etc. (older plants)	150,000
IV	Hard brown coal	
	Lurgi-Spülgas process	
	Sudetanländische Treibstoffwerke, Most (Brüx)	1,000,000
		2,633,000

1/ Approximate equivalent production in U. S. gallons may be found by multiplying by 250. This factor is derived by dividing the weight of 1 metric ton, expressed in avoirdupois pounds, 2.204.62, by 8.829, which is the weight, in avoirdupois pounds, of a U. S. gallon of low-temperature tar of 1.06 average specific gravity.

During the years immediately following World War I, Lurgi Gesellschaft für Wärmetechnik m.b.H., Frankfurt, Germany, developed a carbonizing device in which circulating gases penetrate the coal charge as heat carriers. This device, now called the Lurgi-Spülgas retort, was described in 1936 as follows:

> The low-temperature carbonization oven by the Lurgi*** has the following features: Subdivision of the operation inside of the oven into three distinct zones: (1) Complete drying of the fuel, (2) low-temperature carbonization, and (3) cooling of the semicoke. An even effect of the low-temperature

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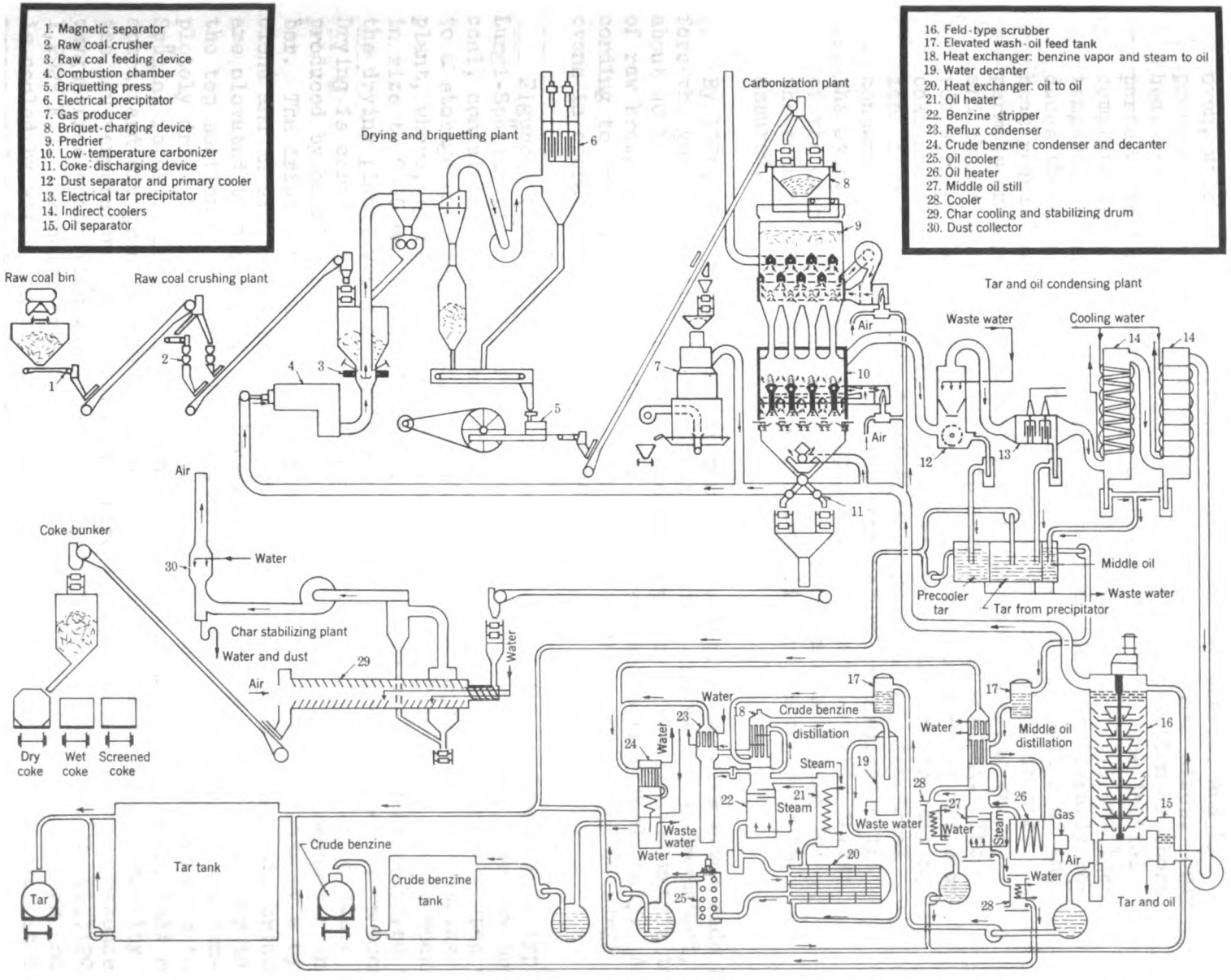

Figure 1. - Brown coal, low-temperature carbonization plant for production of coke, tar, and crude benzine at Offleben, Braunschweigische Kohlen Bergwerke, Helmstedt.

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carbonization is assured by a very uniform distribution of the heat-carrying gases over the whole square-sectional area of the oven, which is vertically subdivided into two shafts, and by preventing the fuel from separating into particular sizes. Only heat-resisting materials are used for the construction of those parts where higher temperatures are applied.***. The initial complete drying of the fuel very much eases the subsequent low-temperature carbonization and at the same time considerably reduces the amount of obnoxious effluents. The reduction of the heat requirements in the low-temperature carbonization zone permits employing low velocities of the heat-carrying gases, and thus a tar is recovered with very low dust content. The recovery of the larger portion of the sensible heat of the semi-coke and the utilization of the waste gases leaving the drying zone down to the point of saturation effect a very low total consumption of heat for the low-temperature carbonization in the oven. By avoiding the application of movable parts inside of the oven and by an exclusive use of ceramic materials in the zone of high temperatures, a long life of these ovens is assured.

By 1936, when the above statement by Dr. Ing. F. A. Ostken was made before the general meeting of the Verein Deutscher Ingenieure at Darmstadt, about 40 Lurgi ovens, each having a daily capacity of 350 to 450 metric tons of raw brown coal, were in operation or under construction in Germany. According to information obtained from another source, there were 62 Lurgi ovens in operation by the first of 1938.

Figure 1 (frame 773) shows the arrangement of the equipment in a typical Lurgi-Spülgas plant used for the carbonization of brown coal. The raw brown coal, containing about 54 percent moisture, is transported from the open mine to a storage and feed bunker, from which it is transported to a crushing plant, where, by means of roller screens and "Titan" breakers, it is reduced in size to 0-20 mm. (0-0.7874 inch). The fine raw coal is next conveyed to the drying plant, where its moisture content is reduced to about 15 percent. Drying is carried out by feeding the fine wet coal into combustion gases produced by burning a portion of the carbonization gas in a combustion chamber. The dried coal recovered from the combustion gases by means of a cyclone and an electrical precipitator is briquetted by a press. The briquets are elevated to the top of the low-temperature retort, where they drop into the top section of the retort or drying oven. Here the briquets are completely dried by combustion gases generated by the burning of gas from the Spülgas retort. In the drying oven the briquets, on losing water, fall mostly apart, and dry granular particles are formed, which move down by gravity into the low-temperature oven, where carbonization takes place in the presence of oxygen-free combustion gases having a temperature of 600°-700° C. (1,112°-1,292° F.). Granular coke is discharged from the oven through a lock and is cooled by water and air in a revolving cylinder. From the latter it is transported to bunkers, from which it is loaded into cars.

The combustion gases leaving the carbonizing oven pass first through a scrubber, where water sprays and a rotating screen remove dust and heavy tar.

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The gases then pass through an electrical precipitator, which removes light tar. The light and heavy tar are combined and shipped to hydrogenation plants. The tar-free gases from the electrical precipitator pass through indirect coolers, which condense middle oil, from which the head and tail fractions are removed in a middle-oil still. The head fraction is combined with the benzine vapors coming from the wash-oil still, whereas the tail fraction is added directly to the tar. By this predistillation of the middle oil before its middle product joins the recirculating wash oil ahead of the Feld washer, good washing is maintained and virtually all of the remaining condensible liquids are removed from the carbonization gas. The wash oil, partly saturated with crude benzine after leaving the Feld washer, is distilled in a benzine stripper and is then recirculated back to the washer. A certain portion of the used wash oil is discharged to the tar. The crude benzine recovered is partly refined and partly hydrogenated to motor fuel.

Waste water leaving the plant at various points contains substantial quantities of phenols, which are recovered at some of the low-temperature carbonization plants.

The coke is screened into suitable sizes for industrial and domestic purposes. The fine material is used in boiler plants and in the Winkler gasification process, the middle size mainly in gas producers, and the large size as household fuel and in central heating plants.

Information concerning the capacity, yields, and other pertinent information for a typical plant of this type at Offleben, near Helmstedt, Germany, is given in Part II in the report covering a visit to the plant of Braunschweigische Kohlen Bergwerke (see page 62).

Krupp-Lurgi Process: Frames 7-9; 248-253; 389-390; 508-509; 514-524; 565; 575-579; 581; 972.

The Krupp-Lurgi process was developed shortly after World War I by Friedrich Krupp A. G. A pilot plant was built at the Amalie plant of Friedrich Krupp near Essen, and later it was rebuilt on a commercial scale. Further modifications with increased capacity were made during World War II.

Similar development of the same process took place in the Saar. An experimental plant was installed by Saargruben A. G. at its Heinitz plant near Neunkirchen (Saar). Later, the same company erected a commercial plant at Velsen.

In the Krupp-Lurgi process as carried out at Wanne-Eickel, Ruhr, and at Velsen, Saar, weakly caking bituminous coal was carbonized in steel ovens heated by recirculated combustion gases. Each oven consisted of six vertical carbonizing chambers 3.1 m. (10.2 feet) long, 2.1 m. (6.9 feet) high, 76 mm. (3 inches) wide at the top, and 100 mm. (3.9 inches) wide at the bottom, set parallel to each other, with spaces between, which comprised the flues through which hot combustion gases were circulated. Each oven was provided with a top cover, a bottom closure, and a grid. The latter was used to close the bottoms of the carbonizing chambers during the charging and carbonizing

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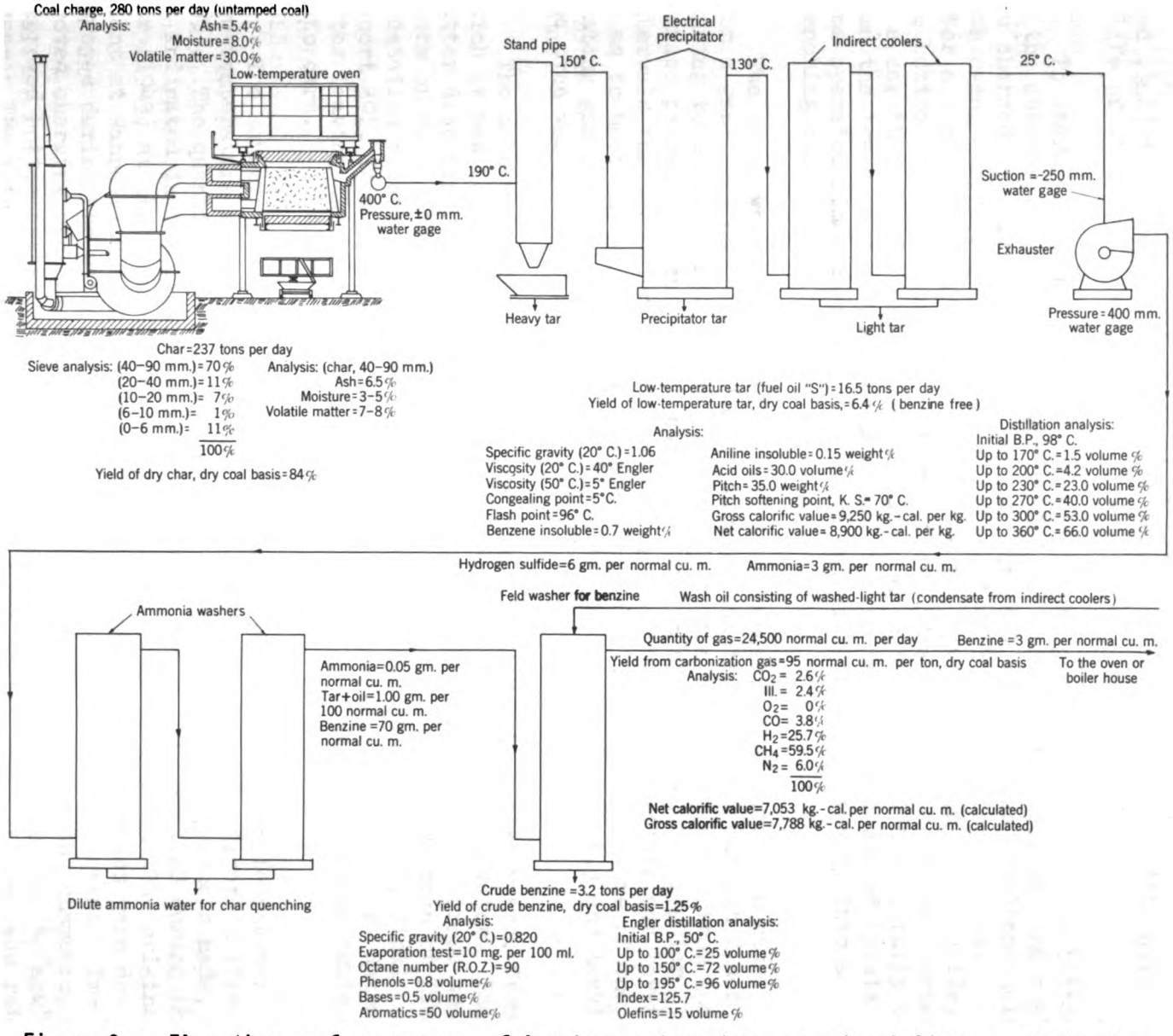

Figure 2. - Flow diagram for recovery of low-temperature tar, ammoniacal liquor, and benzine from low-temperature carbonization plant, Krupp-Lurgi.

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periods. The grid was so arranged that it could be moved horizontally to close or open the bottoms of the coking chambers. The top and bottom covers were water-sealed when in use, so that no gas could escape from the oven. The ovens were constructed entirely of boiler-plate steel. They were in good condition after several years of operation and were expected to have a life of at least 10 years.

To charge an oven, the grid was moved into position to close the bottoms of the chambers, the top cover was rolled horizontally to one side, and coal was charged by gravity from a larry car into all six chambers simultaneously. Each oven, comprising six cells, was charged with about 3 tons of coal. After a carbonizing period of 6 hours the top cover was again rolled aside, the bottom cover was lowered by means of an hydraulic-jack mechanism mounted on a car that could be moved to one side, the grid was moved horizontally to open the lower ends of the coking chambers, and the coke was pushed by six rams operated simultaneously from above and discharged by gravity into a quenching car.

The ovens were heated in the following manner: The combustion gases from a gas-fired combustion chamber were circulated by a fan through flues running to and from four ovens. In each oven the combustion gases from the furnace passed between the chambers and back to the furnace. At regular intervals the direction of the combustion gases through the flues was reversed so as to heat the walls of the chambers uniformly. The temperature of the heating gas entering the oven was 610°-620° C. (1,130°-1,148° F.) and leaving the oven was 560°-580° C. (1,040°-1,076° F.).

The coke was discharged from the quenching car onto a coke wharf, from which it was conveyed by a vibrating conveyor to an inclined elevator. The latter discharged it into a breaker of novel design. This device broke the slabs of coke into small pieces without producing a large proportion of fines. A detailed description of the coke breaker is given in B. I. O. S. Final Report 391, Item 30 (PB 34,738), "A New Coke Breaker", 1946, by C. H. Noton. After leaving the breaker, the coke was lowered by a movable loading chute into cars.

The manner in which the gases from the ovens were handled to recover low-temperature tar, ammonia liquor, and benzine is shown in figure 2 (frame 565). The quantities of coal carbonized and coke and other products made, as indicated in the flow diagram, apply to 24 new ovens installed during the war (1943) at the Wanne-Eickel plant of Krupp Treibstoffwerke. The original plant at Wanne-Eickel, built in 1937, had 32 ovens, some of which were destroyed during the war. They differed somewhat from the later ovens. Improved charging mechanism, grates for closing the bottoms of the chambers, improved bottom covers, and other changes were incorporated in the 24 "new" ovens. The total yearly capacity of the Wanne-Eickel plant in 1943 was rated, in metric tons, as follows: Semi-coke, 204,000; fuel oil, 1,200; and motor fuel, 1,920.

More complete descriptions of the Krupp-Lurgi installation at Wanne-Eickel are given in the following reports: C. I. O. S. File XXV-25 (PB 288),

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"Krupp Treibstoff Werke G. m. b. H., Wanne-Eickel - Fuels and Lubricants", 1945, by C. C. Hall and A. R. Powell; C. I. O. S. File XXXII-91 (PB 4,328), "Lurgi Gesellschaft für Wärmetechnik, Lurgi Haus, Frankfurt-am-Main", 1945, by H. Hollings and J. G. King; and C. I. O. S. File XXXI-30 (PB 4,325), "Krupp-Lurgi Low-Temperature Carbonization Plant of Fried. Krupp A. G. at Wanne-Eickel near Bochum, Germany", by H. H. Lowry and H. J. Rose. Further details also may be found in Part II of the present paper, covering the visit to Fried. Krupp Akt., Essen, and Krupp Treibstoffwerke G. m. b. H., Wanne-Eickel. A complete description of the Kurpp-Lurgi process is given in Bureau of Mines Information Circular 7395.

Geissen Process: Frames 3; 507-508; 971.

During the years immediately following World War I, two new processes for carbonizing brown coal were introduced in Germany. One of these was the Lurgi-Spülgas process described above, in which carbonization was effected by direct contact between the brown coal and recirculated hot combustion gases. The other was the Geissen process. The arrangement of the metal retort used in the Geisson process is illustrated in figure 3 (frame 507). Operation is as follows: Brown coal is dried to approximately 15 percent moisture (from 54 percent) in a separate drier. It is then carbonized in the Geissen oven while passing downward between a revolving corrugated cylinder (made of a special iron alloy) and stationary annular collars. This results in a continuous turning of the pieces of fuel as they pass downward in a thin layer and immediate withdrawal of the low-temperature carbonization products through offtakes for primary gas and char.

The retort is heated by passing combustion gases from a central gas burner between the external wall of the burner and the internal wall of the revolving corrugated-iron cylinder. Each retort of this type has a capacity of about 150 metric tons of raw brown coal (54 percent water) per day. Up to 1938, about 35 units of Geissen ovens had been installed in Germany. Several of them were still operating during World War II, but no new ones had been built after 1938.

Borsig-Geissen Process: Frames 3; 224-230; 384-385.

In 1935, Borsig modified the Geissen low-temperature oven and introduced the Borsig-Geissen oven or retort. Its arrangement is illustrated in figure 4 (frame 224). Instead of using a corrugated, revolving cylinder made of heavy iron castings, the Borsig-Geissen retort used a plain stainless-steel tube for the rotating member. The burner was situated at the top of this member, and the combustion gases, after passing downward through the rotating cylinder, passed upward through a jacket surrounding the retort. Partly dried brown coal introduced at the top passed downward between the rotating cylinder and the stationary baffles, as in the old Geissen oven. The coal charged to the retort must not be larger than 1/2-inch and must not contain more than 10 percent of fines below 1/25-inch size. The char was discharged from the bottom of the oven. Part of the oven gas was used for heating the retort; the remainder was withdrawn from the side.

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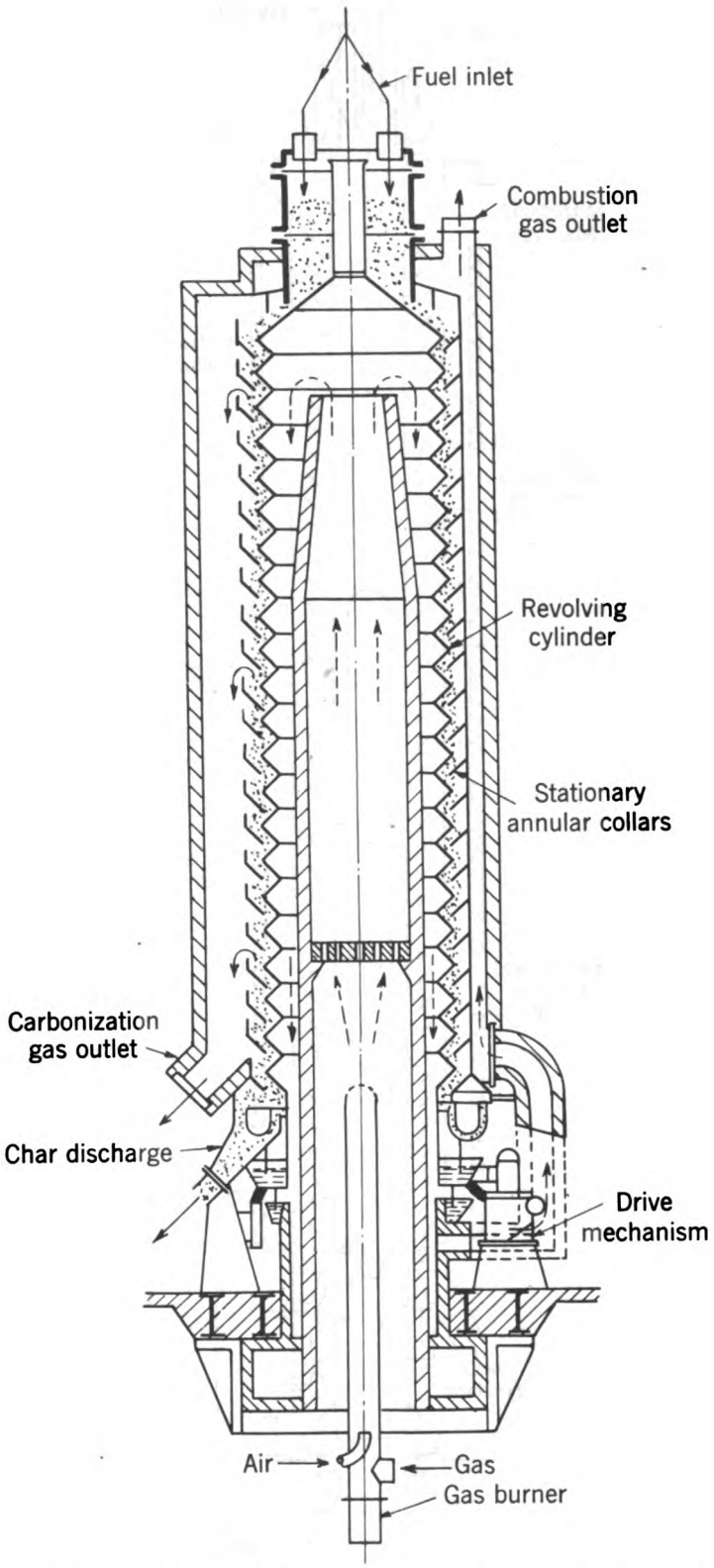

Figure 3. - Geissen vertical revolving retort for low-temperature carbonization of brown coal, Kohlenveredlung und Schwelwerke A.-G., Berlin.

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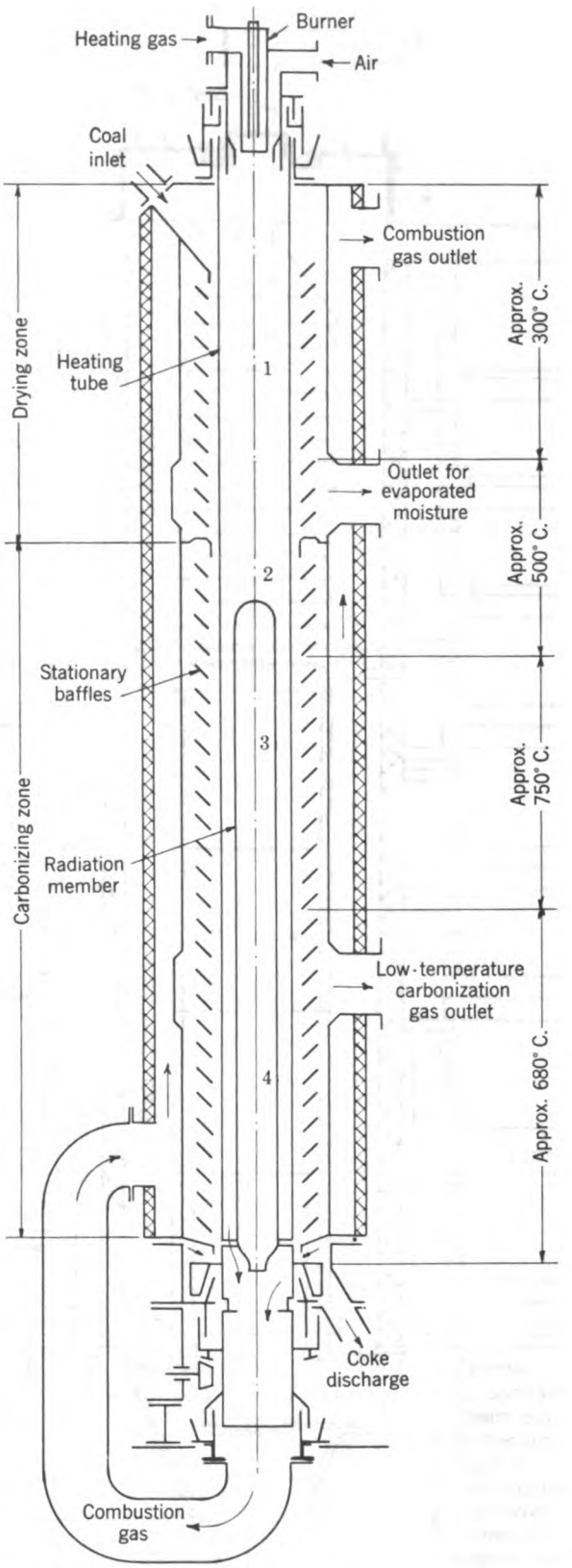

Figure 4. - Vertical cross section of Borsig-Geissen low-temperature carbonization retort.

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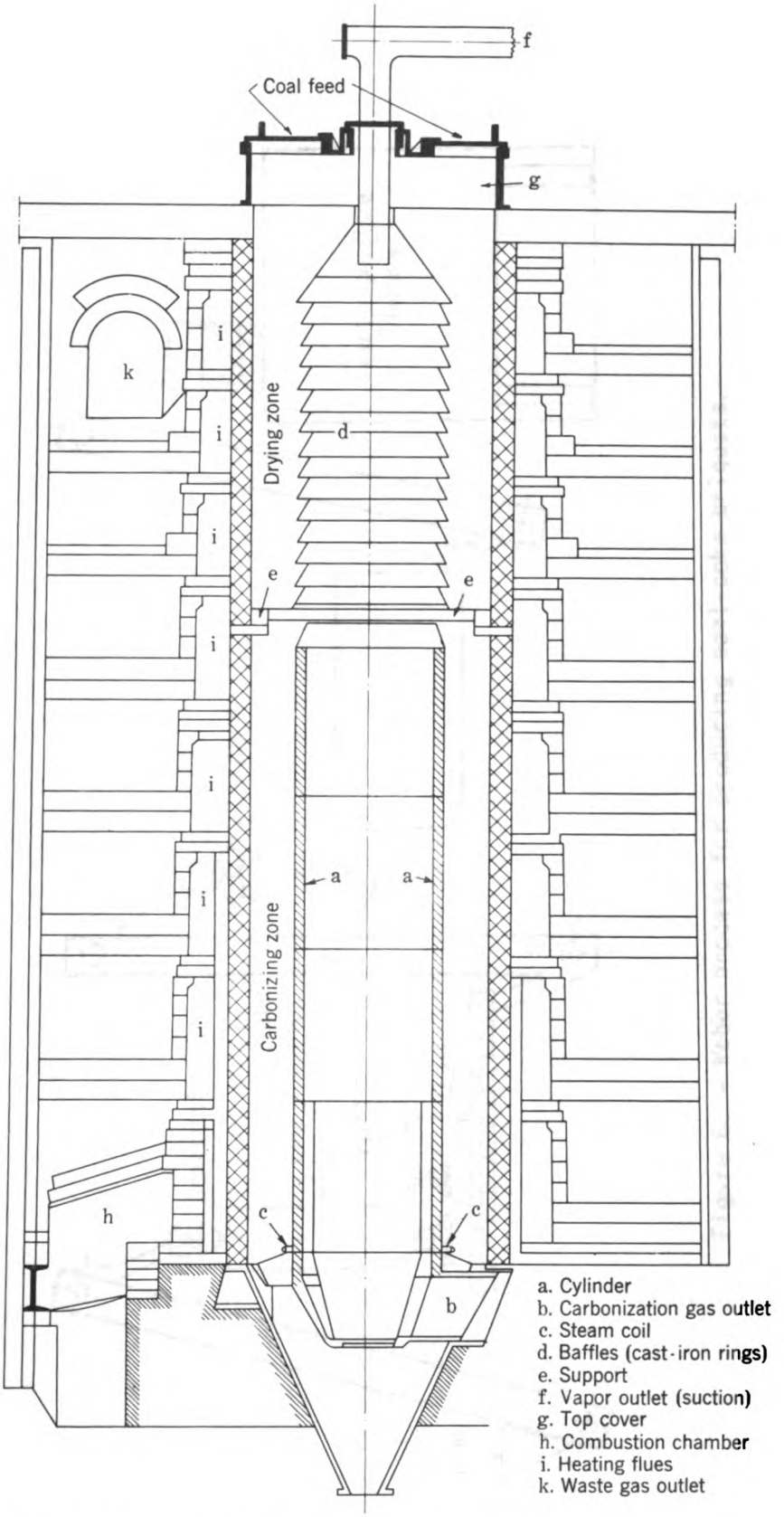

Figure 5. - Modern Rolle retort for low-temperature carbonization of brown coal.

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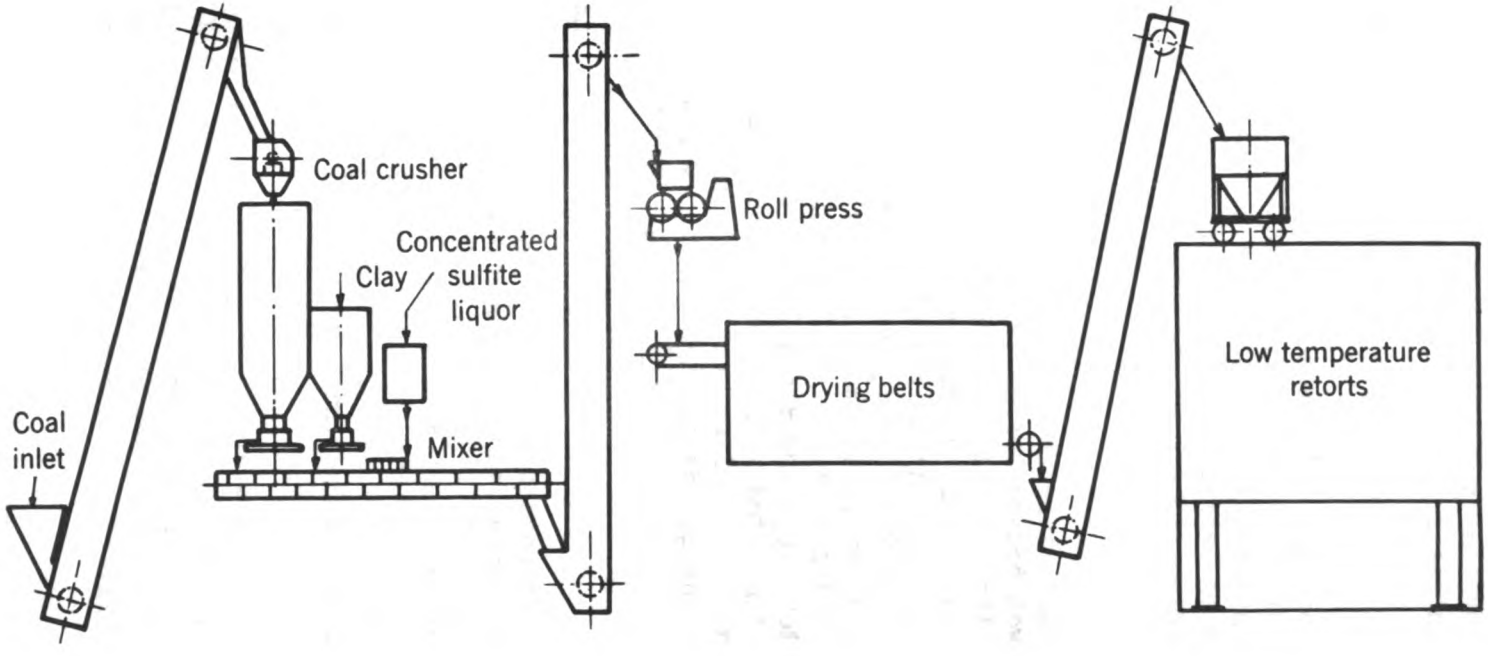

Figure 6. - Weber process for producing semi-coke briquets.

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Besides four test ovens at different places, only one commercial plant, belonging to the Leipziger Braunkohlenwerke at Kulkwitz, was built in Germany. This plant had operated since 1937, but in 1944 only a few of the 20 retorts were in operation because of the lack of stainless cast steel.

Rolle Process: Frames 3; 222-224; 335-339; 507.

A vertical oven of circular cross section for the low-temperature carbonization of brown coal was developed by Dr. Rolle in 1872. Many modifications were made in attempts to increase its efficiency, but, owing to low capacities (3-15 tons per day) and low tar yields, no new units were built after about 1924, when Geissen introduced his all-metal oven with the corrugated, vertical, revolving drum. It is reported that there were 588 Rolle ovens in Germany in 1938.

One arrangement of the Rolle oven is shown in figure 5 (frame 223). The partly dried brown coal to be carbonized passed downward in the annular space between a central metal cylinder and an external ceramic cylinder. Heating of the coal was effected by heating flues arranged around the exterior of the ceramic cylinder. In the older model of the Rolle retort, the inner cylinder consisted of overlapping, narrow, conical, cast-iron rings. In the newer model (fig. 5, frame 223), the upper one-third of the inner cylinder was constructed in this manner, but the lower two-thirds consisted of plain, cylindrical, cast-iron rings (a). Moisture liberated in the upper zone was withdrawn through the opening between the overlapping cast-iron rings (d) and left the retort from outlet (f). In the lower zone, the carbonization gases were withdrawn through the plain cast-iron cylinder (a) and left the retort through opening (b).

Related Processes:

Mention is made at this time of two processes that were not included in the preceding list of "Low-Temperature Carbonization Plants Operating in Germany in 1943-1944" (see table 1, page 12). They are mentioned here, however, because each of them was related, at least indirectly, to the low-temperature carbonization industry. The two processes are, respectively, the Weber and the Pintsch or Pintsch-Weber. The following discussions of these processes are based upon information furnished by Adolf Thau of Didier Werke, A. G., Berlin.

Weber Process: Frames 26-28; 58. - Weber has developed processes in Germany for the briquetting of semi-coke and for the production of briquets from noncaking bituminous coal, which are subsequently subjected to low-temperature carbonization to obtain tar and smokeless fuel. The operation of one of Weber's processes of the latter type is illustrated in figure 6 (frame 58).

The coal is carried by an elevator to a crusher, from which it drops into a feed tank. From the latter it passes to a mixer, where a suspension of clay and concentrated sulfite liquor from pulp plants are combined with it in proper proportions by heating and vigorous agitation. The mixture then

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passes to a roll press, where briquets are formed. They are dried on slowly moving belts until most of the water has been expelled. They are then subjected to low-temperature carbonization to produce "very reactive, dense, hard, and smokeless-burning briquets or ovoids."

According to Dr. Thau, "this process, though old in itself, has been developed further, and it now forms the bridge over which metallurgical coke may be manufactured from noncaking coals and even from brown coal. There is no doubt that not only in Germany, England, and France, but also in the United States, a distinct shortage of good-coking coal makes itself more and more acutely felt, while noncaking coals rich in volatile matter are available in abundance."

Further possible application of Weber's processes to the briquetting of iron ore is discussed by Thau, as follows:

> Weber, who has in the meantime made use of every suitable binding medium which offers the greatest economy, has developed a further process which is likely to have a great effect upon the present mode of pig iron manufacture. There is no doubt that the richest iron ores on the Continent as well as in England and in the United States contain large proportions of smalls, and others are so soft that the blast furnace can be burdened with them only if they are briquetted first. In any case the operation of the blast furnace is always hampered by the iron ore dust partly kept in suspension by the blast furnace gas and partly being deposited where it is not wanted and frequently gives rise to serious troubles.
>
> Weber developed, based upon his former research work on briquetting, a process which is to manufacture pig iron without using coking coal or metallurgical coke and utilizing at the same time small-grained or soft-iron ores. To accomplish this Weber mixes finely-ground noncaking coal and powdery ore in a certain proportion, adds a binder, such as, for instance, pitch, oil residuals, or partly hydrogenated coal, and transforms the mixture by roller presses into ovoids, which are subsequently subjected to low-temperature carbonization to recover the primary tar. By distilling the latter, pitch for binding the next batch of ovoids is being recovered. The carbonized ovoids can be used as a fuel for liquid-slag gas producers or for low-shaft furnaces and in both cases a pig iron of good quality is being produced at the same time. The carbon contained in the ovoids suffices to effect a chemical reduction of the iron ore. If there is a large excess of carbon, the furnace or gas producer may be burdened in addition with a corresponding proportion of lumpy iron ore.
>
> The large volumes of gas produced may be utilized for synthetic purposes, and Weber further suggests a combination with a coal-hydrogenating plant in order to improve the economical results of the latter by way of using the coal which has been partly hydrogenated.

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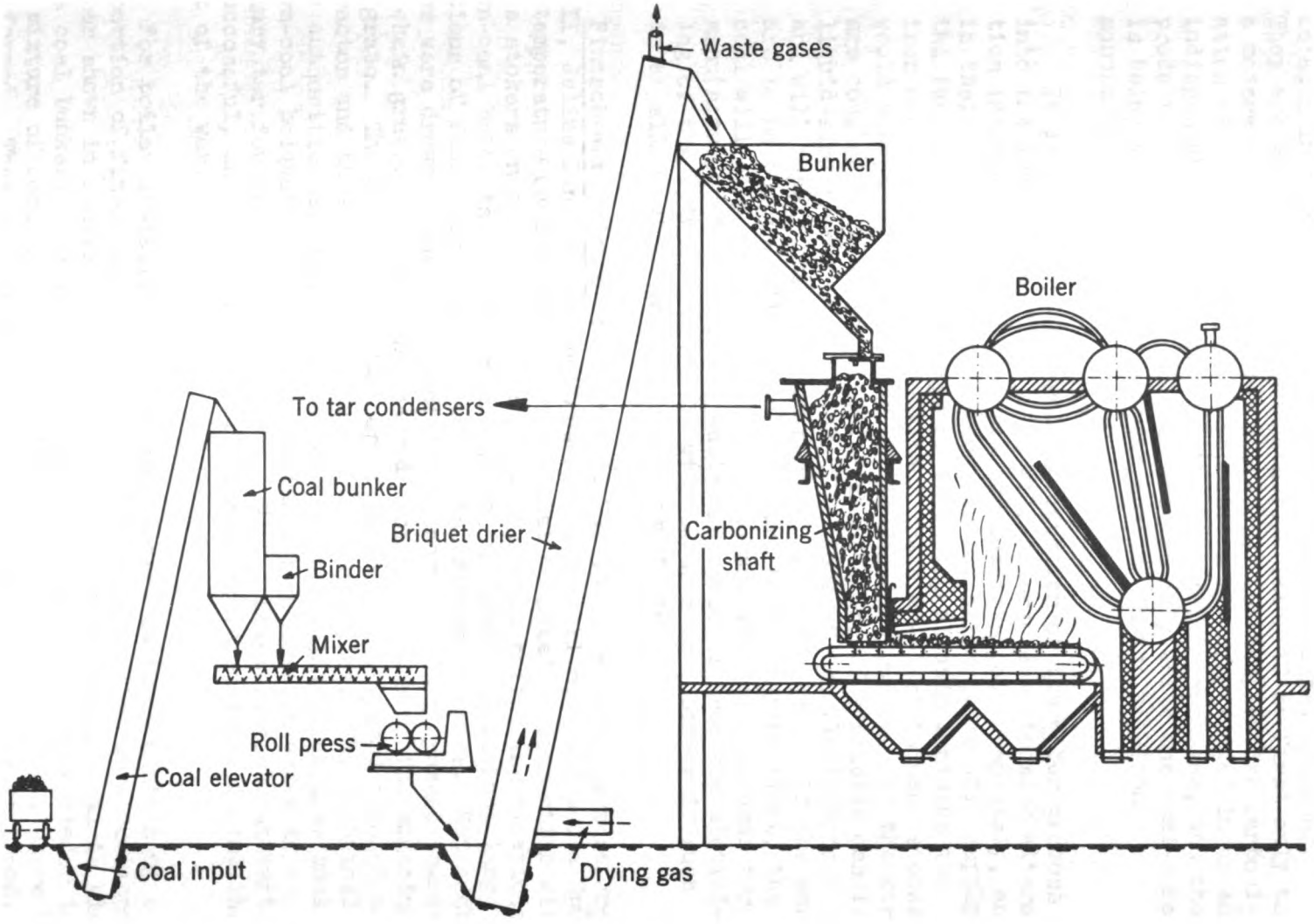

Figure 7. - Pintsch-Weber low-temperature carbonization process combined with steam-boiler plant.

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It would exceed the scope of this report to go into further details in connection with the processes developed by Weber. They are sure to be realized sooner or later if at first only to a moderate extent. By these processes, low-temperature carbonization will get on to a sound foundation by being wedged in as an indispensable bridge between other industrial processes, and the products recovered will be oils and gas only while the semi-coke is being worked up together with the ore in the pig-iron manufacture.

If it comes to a general introduction of the Weber process into the iron works, their face will undergo an extensive alteration inasmuch as the coking plants will disappear completely, and in their place a low-temperature carbonization plant will supply the fuel which, in the shape of briquets, already contains the iron ore in the right proportion. The high-blast-furnace stacks would disappear to make room for low-shaft furnaces. If the works are coupled with a plant for the manufacture of synthetic gasoline, liquid-slag producers will take the place of the blast-furnace stacks and will supply the gas necessary for the synthesis. If the works are to be coupled with a high-pressure hydrogenating plant, the coal will only partly be hydrogenated and will then be used for manufacturing the briquets, while the residual from the distilling of the hydrogenation oils will be used as a binder if such is at all required under these circumstances.

Pintsch-Weber Process: Frames 21-23; 53. - Several years before World War II, Julius Pintsch Kommanditgesellschaft, Berlin, developed a Spülgas low-temperature carbonization process that operated in connection with chain-grate stokers on boiler installations. Numerous boiler installations using brown-coal briquets or bituminous, noncaking lump coal without large proportions of fines were equipped to use the Pintsch process. Hot combustion gases were drawn through the coal in the magazine as it passed downward to the chain grate. Hot semicoke passed from the bottom of the magazine onto the grate. The gas leaving the top of the magazine passed through a tar extractor and then went to a burner under the boiler. The technical military authorities attempted to have all boilers using bituminous coal or brown-coal briquets equipped for the Pintsch process in order to recover primary tar for use as fuel oil by the Navy. However, these attempts were unsuccessful, and no boiler installations were so equipped during the latter part of the war.

For boiler installations that must depend on fuels containing a large proportion of fines, the processes of Pintsch and Weber were combined in the manner shown in figure 7 (frame 53). The fine bituminous coal is elevated to a coal bunker, from which it drops into a mixer, where a binder is added. The mixture of coal and binder is then fed into a roll press, where briquets are formed. They are conveyed by a slow-moving elevator with broad, perforated buckets to a bunker. Hot gases introduced into the housing of the elevator at the bottom escape at the top and harden the briquets as they are conveyed upward to the bunker. From the bunker, the briquets slide into a

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carbonizing shaft, where they are converted to semi-coke before discharging onto the chain grate of the boiler. The hot gases are drawn upward through the carbonizing shaft as the briquets pass downward. Tar leaving the carbonizing shaft with the hot gases is removed by means of tar extractors.

Low-Temperature Carbonization Plants under Construction at End of World War II

In addition to the plants described above that were actually operating when the war ended, other plants were under construction and still others were in the planning stage. The following plants were under construction:

Heinrich Koppers plant at Oheim mine of Bergwerksverwaltung Oberschlesien, Katowice (Kattowitz) - continuous, vertical, ceramic ovens with a capacity of 1,000,000 metric tons per year of weakly caking, bituminous coal and estimated tar production of 92,000 metric tons.

Didier Werke plant at Oswiecim (Auschwitz) - 24 chambers, 550 metric tons per day of noncoking coal, or 200,000 metric tons per year.

Brennstoff Technik plants at Marienau by Bergwerksverwaltung Kleinrosseln, Forbach, Lorraine (Lothringen) - (1) 16 ovens, 500,000 metric tons per year of bituminous-coal briquets, half-completed at end of war, and estimated tar production of 50,000 metric tons; and (2), the same as (1), construction just started at end of war.

Brennstoff Technik plant at Jawischowitz by Bergwerksverwaltung, Katowice (Kattowitz), Upper Silesia - 500,000 metric tons per year of bituminous nut coal, construction started, and estimated tar production of 42,500 metric tons.

Following are brief descriptions of the processes employed by Koppers, Didier Werke, and Brennstoff Technik, respectively.

Heinrich Koppers Process: Frames 556-564.

When the war ended, a Koppers low-temperature carbonization plant was under construction at the Oheim mine of Bergwerksverwaltung Oberschlesien, Katowice (Kattowitz), in Upper Silesia. It was to be used for coking an Upper Silesian high-volatile bituminous nut coal.

The dimensions of each oven, in millimeters, were as follows: Total chamber height, 11,500; length of chamber, 3,500; width of chamber at top, 250; width of chamber at 5,700 mm. below the top, 350; and width of chamber at bottom, 350. Equivalent dimensions in feet are 37.7, 11.5, 0.8, 18.7, 1.1, and 1.1, respectively.

The oven walls were lined with fire brick, but the heating flues were constructed of silica. By means of automatic reversing equipment, the heating gases were to pass alternately upward and downward through the vertical heating flues and through the upper and lower regenerators. The

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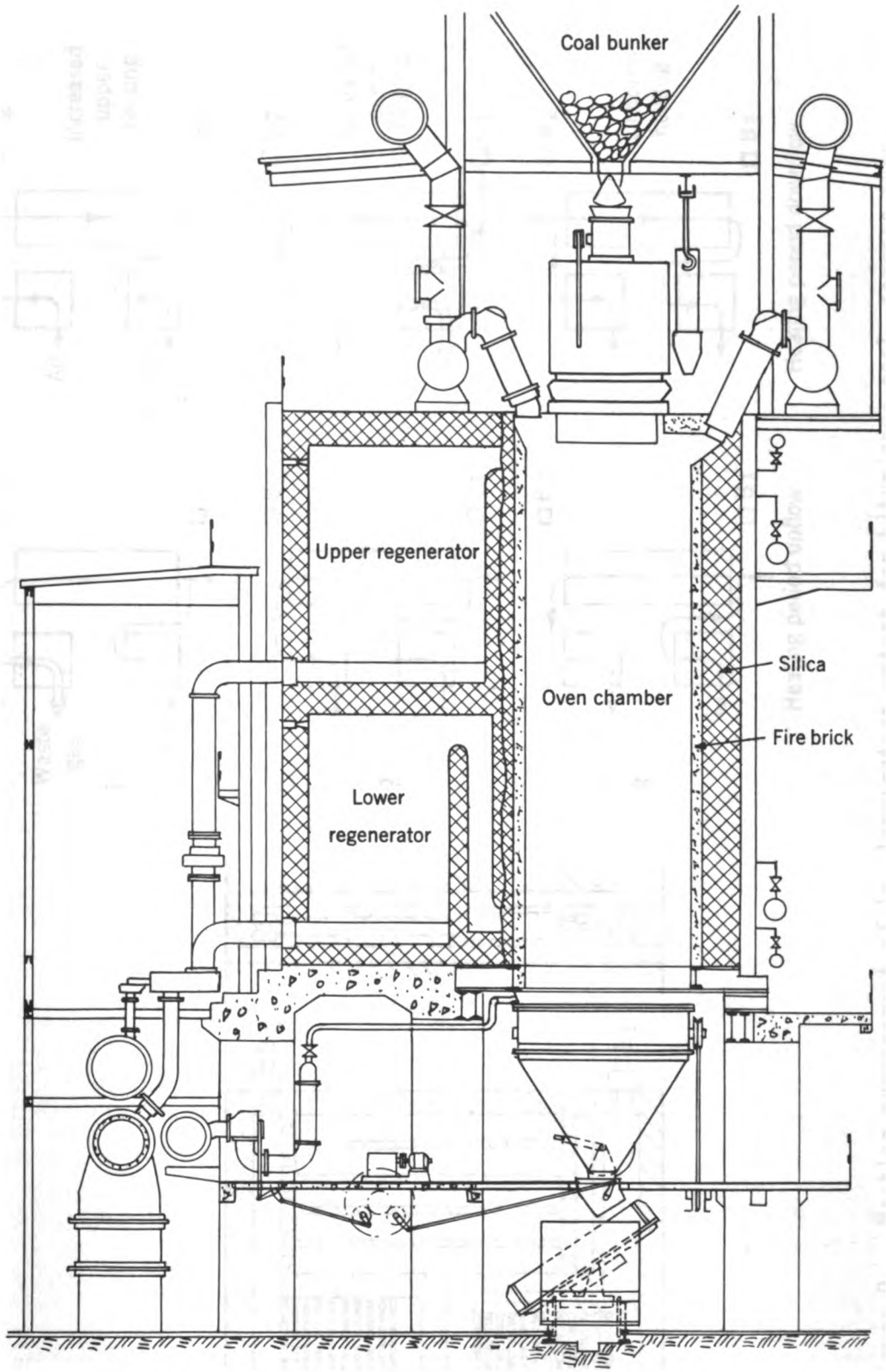

Figure 8. - Low-temperature retort for bituminous coal, Koppers type.

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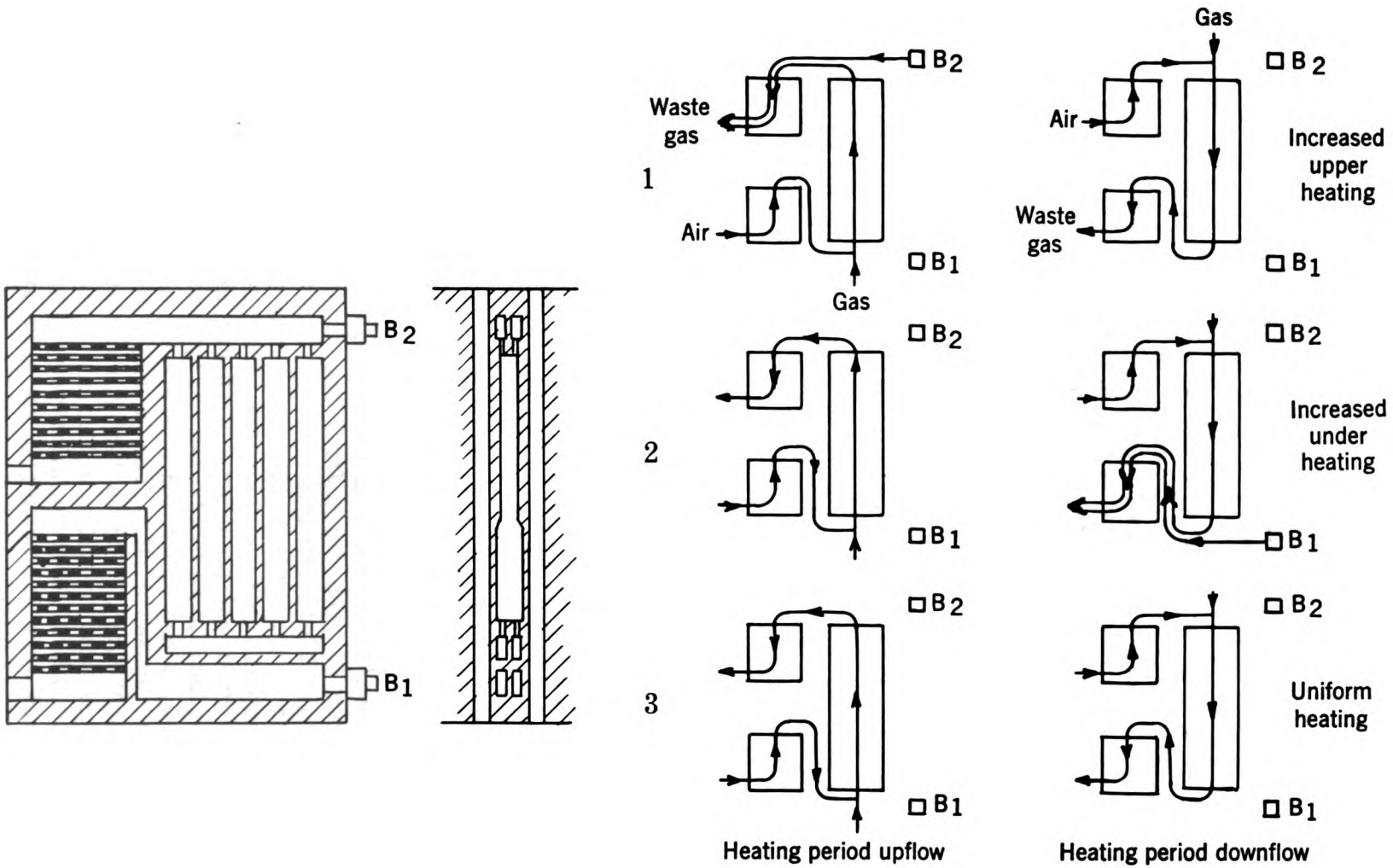

Figure 9. - Heating arrangement of low-temperature retort for bituminous coal, Koppers type.

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arrangements of the flues and regenerators with respect to an oven chamber are shown in figures 8 and 9 (frames 563 and 564). Figure 9 also shows diagrammatically how, by means of auxiliary burners, additional heat could be supplied to the upper and lower regenerators as desired in order to increase the temperature in the upper or lower part of the chamber or maintain a uniform temperature over its entire height.

Above each unit of 15 ovens was a coal bunker from which coal dropped into the feed hopper on the top of each oven chamber. From the feed hopper the coal dropped into the carbonizing chamber and the coke was discharged into a bottom hopper, from which it was withdrawn into a tilting cart, which transported it to a special quenching station.

In order to precool the coke entering the discharging mechanism, a portion of the gas from the chambers, free of tar and benzine, was returned to each chamber near the bottom and just above the coke-discharge outlet. Also, water injected into the coke produced steam, which mixed with the recirculated gas and passed upward through the coke in the chamber, where water gas was formed in addition to low-temperature carbonization gas.

The ovens or chambers were to be heated either by part of the oven gas or by gas produced in generators from coke of 3-20 mm. (about 1/8-3/4 inch) size.

The flow of the gas from the ovens was as follows. Leaving the ovens through two standpipes, it passed through collector mains to indirect coolers, followed by mechanical or electrical tar separators.

Removal of free ammonia was effected in the final cooler, from which the gas passed to the benzine-absorption equipment and then to holders. A part of the gas, as indicated above, was returned to the bottoms of the ovens for precooling the coke.

The operating results when carbonizing Upper Silesian, high-volatile, bituminous, nut coal containing 5 percent moisture and 7 percent ash and making coke with 3 percent volatile matter and gas with a gross calorific value of 4,500 kg.-cal. per normal cubic meter (or 470 B.t.u. per cubic foot at 60° F. and 30 inches Hg pressure, saturated), were expected to be as follows: Coal throughput per chamber per day, 25 metric tons; coke yield (3 percent moisture content), 64 percent; gas yield per metric ton of coal, 300 normal cubic meters; gross calorific value of gas per normal cubic meter, 4,500 kg.-cal.; gross calorific value of gas per kilogram of coal, 1,250 kg.-cal.; low-temperature tar yield, 9.2 percent; low-temperature benzine yield, 0.8 percent; and heat consumption per kilogram of coal, 550 kg.-cal. Equivalent English units are: Gas yield per short ton (2,000 pounds) of coal, 10,330 cubic feet at 60° F. and 30 inches Hg pressure, saturated; gross calorific value of gas per cubic foot at 60° F. and 30 inches Hg pressure, saturated, 470 B.t.u.; gross calorific value of gas per pound of coal, 2,250 B.t.u.; and heat consumption per pound of coal, 990 B.t.u. The combined tar and benzine yield amounted to 87 percent of that found by the Fischer assay.

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Some of the advantages claimed by Heinrich Koppers G.m.b.H. for this type of equipment were as follows:

1. Low-temperature processes using iron retorts must operate at or below 600° C. (1,112° F.), avoid local overheating, and use relatively small chambers. These precautions are not required in the Koppers process.

2. Low-temperature carbonizing processes using circulating combustion gases for heating the coal produce tar and gas of inferior quality, as compared with tar and gas produced in the Koppers process.

3. The gas yield is greater in the Koppers process, and stronger coke is produced, owing to the higher temperatures employed.

4. By recirculating a portion of the oven gas to the carbonizing chamber, the efficiency of carbonization is raised 50 percent above that of processes in which no gas is recirculated.

5. The coke produced in the Koppers process is more completely devolatilized than coke from iron retorts, but its reactivity is high.

Prior to the receipt of the order in 1942 for the installation at Oheim mine, the Heinrich Koppers Co. had installed plants of this type for gas production in gas plants, for calcining petroleum coke (Erftwerke A. G. Grevenbroich), for coking ovoid briquets at 700° C. (1,292° F.) (Mines de Carmaux, South France), for coking weakly caking high-volatile bituminous coal at 700° C. (1,292° F.) (Mines de Bruay, South France), and for production of mixed gas (distillation gas plus water gas) from noncaking, high-volatile bituminous, nut coal (Nitrogen Works, Ube, Japan).

Didier Werke Process: Frames 9-13; 31-34.

According to Dr. Thau of Didier Werke, Berlin, he had suggested in 1938 that continuous, ceramic, vertical-chamber ovens be used in Germany for the low-temperature carbonization of noncaking coal. He pointed out that vertical ovens had been employed for this purpose by Woodall-Duckham at the Richmond Gas Works near London, but the chambers were constructed of metal and did not last very long. Similar ovens with ceramic chambers and operated semicontinuously were installed a few years later by the Fuel Research Institute, Greenwich.

Didier Werke, acting on Dr. Thau's suggestion, constructed a trial unit at Stettin for use on a semicommercial scale. Then, according to Dr. Thau, Heinrich Koppers G.m.b.H. altered the design of the continuous vertical-chamber ovens which it was building in Upper Silesia for the manufacture of "reducing char" in such manner as to permit the continuous low-temperature carbonization of noncaking coal.

According to Dr. Thau, Dr. C. Otto & Company then modified its continuous vertical-chamber ovens so as to carbonize noncaking coal continuously at low temperatures and collaborated with Didier Werke and Heinrich Koppers in

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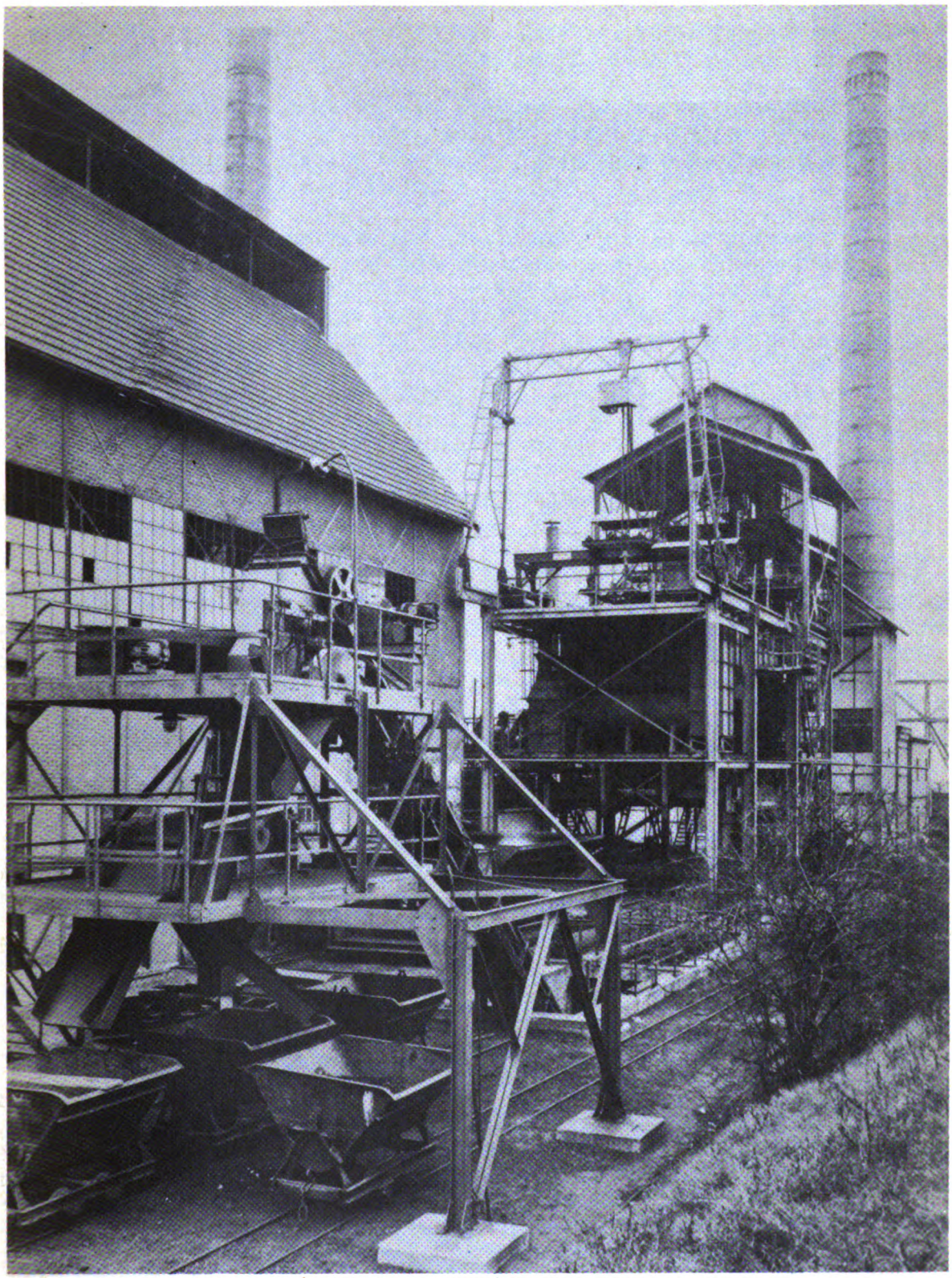

Figure 10. - Experimental low-temperature carbonization installation at Gaswerk Neukölln of Berlin gas works, Brennstoff Technik G. m. b. H., Essen.

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the successful development of continuous, ceramic, vertical-chamber, low-temperature, carbonizing equipment for noncaking coal.

The reason why the same type of equipment is not suitable for the low-temperature carbonization of caking coal in spite of the fact that vertical ovens were designed originally for manufacturing gas coke from caking coal is explained by Dr. Thau as follows:

> In order to penetrate the coal charge evenly and within a reasonable time, the low-temperature carbonizing chamber must be pretty narrow (about 200 mm. or 7.9 inches). Since semi-coke does not contract as much after carbonization has been completed as high-temperature coke, and since the friction set up between wall faces and coke ends would be too great to be overcome by the weight of the charge, the coke charge is sure to stick in the ovens, and for this reason only noncaking coal is treated in these ovens. Owing to the continuous downward movement of the coal charge, the slight caking of coals having such a tendency can easily be prevented by oxidizing the coal before it enters the chamber.

In a commercial plant designed by Didier Werke, the ovens had the following dimensions: Height, 930 mm.; length, 400 mm.; width, 220 mm. at top and 280 mm. at bottom. Equivalent English dimensions are: Height, 3.1 feet; length, 1.3 feet; width 8.7 inches at top and 11.0 inches at bottom. The throughput of the plant, consisting of 24 chambers of this size, was 300,000 metric tons of noncaking coal per year. The arrangement of the Didier Werke oven with respect to heating flues and regenerators is illustrated by frames 31, 32, and 33. This plant was said to have been under construction at Oswiecim (Auschwitz) when World War II ended.

In the Didier Werke equipment, like that of Heinrich Koppers G.m.b.H., recirculated, tar-free, chamber gas was introduced into each chamber, above the coke extractor. By recirculating a larger or smaller amount of gas, the velocities of gases and vapors expelled from the coal could be regulated so that the length of time during which the vapors and gases remained inside the chamber could be adjusted within wide limits, resulting in corresponding adjustments of tar quality and yields.

Brennstoff Technik Process: Frames 8; 97-99; 245-248; 445-449; 486-489; 490-491; 495-506; 668-672.

Brennstoff Technik, Essen, was founded in Germany in 1933 to develop a process for the low-temperature carbonization of coal that involved the use of movable chamber walls. The process was tried first in a small pilot plant in Essen. Between 1935 and 1939, larger-scale tests were made in a plant having a daily capacity of 10 to 12 metric tons of fine coal. This plant was built for Delbrückschächte der Preussischen Berg-und Hüttenbau A. G., Hindenburg, Upper Silesia. A test plant having a daily throughput of 30 metric tons of fine coal or 50-60 metric tons of nut coal or briquets was operated by the Städtischen Gaswerke in Berlin between 1938 and 1944. A picture of this plant is shown in figure 10 (frame 495).

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Full-scale development of the Brennstoff Technik process was planned in 1942, when the German Government announced a program for the low-temperature carbonization of 23,000,000 metric tons of coal per year, of which 14,000,000 metric tons were to be carbonized in Upper Silesia, 6,000,000 in the Ruhr, and 3,000,000 in Lorraine (Lothringen). As a part of this program, four new plants of 500,000 metric tons capacity each were to be constructed. One of these was to be a plant using ceramic chambers. (The order for the vertical, ceramic, chamber oven plant in Upper Silesia was placed by the German Government at that time.)

The other three plants included in the program were to be of the B.T. (Brennstoff Technik) type. An order for Brennstoff Technik to build two of these plants in collaboration with the Mineralöl-Baugesellschaft, Berlin, was received from the Bergwerksverwaltung Kleinrosseln, Forbach, Lorraine (Lothringen, State Plant). They were located at Marienau and were known as Marienau I and Marienau II. Each was to have 16 ovens and be used for the low-temperature carbonization of coal briquets.

The order for the third B.T. unit, also to be built by Brennstoff Technik in collaboration with the Mineralöl-Baugesellschaft, was placed by Bergwerksverwaltung, Katowice (Kattowitz), Upper Silesia (State Plant). This plant was to have 20 ovens and be used for the low-temperature carbonization of nut coal. Its location was Jawischowitz.

When the war ended, Marienau I was half finished, and the construction of the Marienau II and Jawischowitz plants had been started.

The arrangement of an oven of the B.T. type for the low-temperature carbonization of 50 metric tons of fine caking coal per day is shown in figure 11 (frames 487 and 488). The operation of the oven was as follows: Fine caking coal was dropped from an overhead larry car into the carbonizing chambers or spaces between the movable iron heating walls. After carbonization had been completed by heat from combustion gases circulated through the movable walls, the walls were pushed apart by a mechanical device and the coke dropped into a quenching car. The manner in which the movable walls are connected by ducts (which also serve as trunnions) to the inlet and outlet flues for hot combustion gases is indicated in figure 11.

Low-Temperature Carbonization Processes Under Development in Germany at End of World War II

In addition to the low-temperature carbonization processes described above which were actually in use or for which commerical plants were being constructed during World War II, two other processes were being considered or were under development, according to Dr. Adolf Thau of Didier Werke A.G., Berlin. Following are brief discussions of each based upon information received from Dr. Thau. The processes are discussed under the following titles: (1) The Büttner Drier and (2) The Blümner Process.

The Büttner Drier: Frames 14-16; 35; 938-957; 958-961; 962-969; 1159-1199; 1200-1230.

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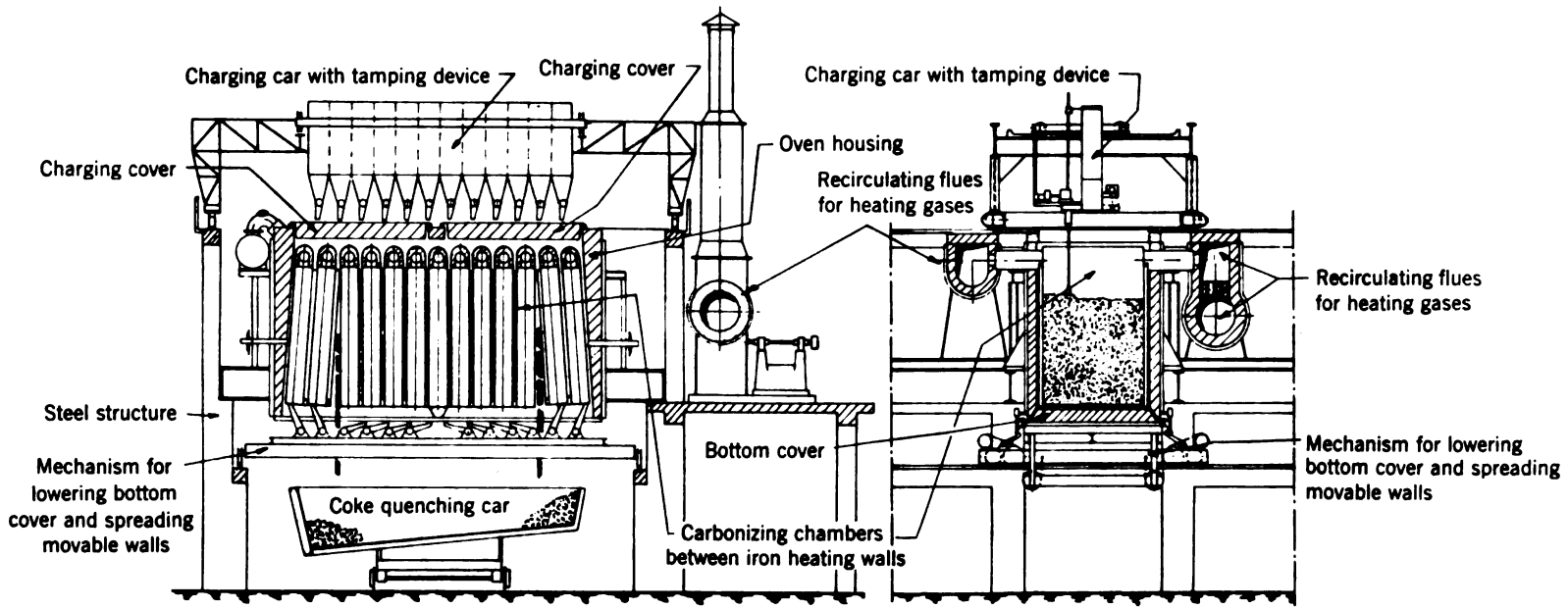
Charging car with tamping device
Charging cover
Charging car with tamping device
Charging cover
Oven housing
Recirculating flues for heating gases
Recirculating flues for heating gases
Steel structure
Mechanism for lowering bottom cover and spreading movable walls
Coke quenching car
Bottom cover
Carbonizing chambers between iron heating walls
Mechanism for lowering bottom cover and spreading movable walls

Figure 11. - Low-temperature oven for carbonization of caking bituminous coal fines (capacity, 50-metric tons per day), Brennstoff Technik G. m. b. H., Essen.

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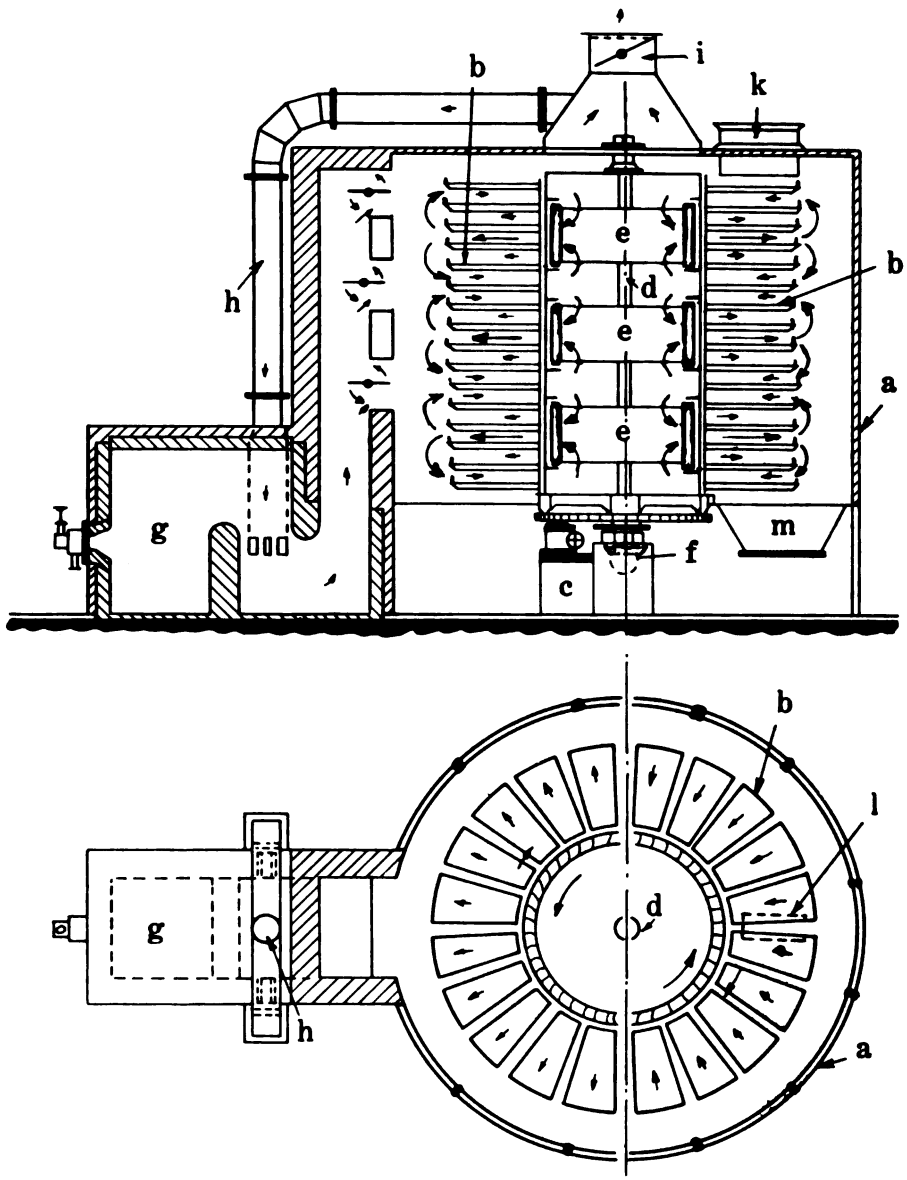

a. Housing
b. Revolving shelf with disc rings
c. Drive for shelves
d. Circulating fan shaft
e. Circulating fans
f. Gear drive for circulating fans
g. Combustion furnace
h. Recirculating pipe for combustion gases
i. Waste gas outlet
k. Coal feed
l. Coal slot
m. Char discharge

Figure 12. - Büttner coal drier adapted to low-temperature carbonization of fine coal.

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The Büttner drier is mentioned briefly here because, according to Dr. Thau, before the end of World War II it was being considered for the low-temperature carbonization of fine noncaking bituminous coal.

Dr. Thau commented as follows concerning the use of the Büttner drier as a low-temperature carbonizing device: "The utilization of noncaking fines, such as floated coal or slimes, oil shale, and all such materials which cannot be treated by the ovens mentioned above (B.T., Krupp-Lurgi, Lurgi-Spülgas, Geissen, Borsig-Geissen, Rolle, Didier, Koppers, and Otto) offered a serious problem, which, however, has in the meantime been solved quite successfully by adapting the Büttner turbine drier for the purpose. The application of the drier to the low-temperature carbonization of fine, noncaking, bituminous coal was contemplated in several places before the end of the war."

Figure 12 (frame 35) shows two views, in cross section, of a Büttner drier arranged for the low-temperature carbonization of fine coal. The following description was furnished by Dr. Thau. A stationary cylindrical housing (a), well-insulated against heat losses by radiation, encloses a structure consisting of circular shelves (b), which are revolved slowly by an electric motor (c). A vertical shaft (d) in the center of the drier supports several circulating fans (e), the blades of which are sloped like those of a turbine. The fans are rotated by means of a gear (f) in a direction opposite the direction of rotation of the shelves. If hot waste gases from some nearby source are not available, hot combustion gases are produced by burning powdered fuel or gas in a furnace (g). The hot combustion gases from the furnace pass into the cylindrical chamber and then are drawn between the shelves by the turbine fans and upward to the top of the drier, where a part of the combustion gas is recirculated back to the furnace through a pipe (h), and the remainder is released to the atmosphere through a butterfly damper (i). The coal to be dried or carbonized enters through a top opening (k). It enters at a uniform rate, so as to be distributed evenly on the top disk or shelf. When the coal has made one revolution on the top shelf it is forced, by means of a stationary adjustable scraper, to drop through a slot (l) onto the next lower-rotating shelf, where the process is repeated. After one revolution on each of the shelves in the drier, the dried coal or low-temperature coke is discharged through a bottom opening (m) onto a belt, which transfers it to the screening plant. The coal is not agitated violently while passing through the drier, and dust formation is small.

Büttner driers are manufactured by Büttner-Werke A.G., Uerdingen-Krefeld. A visit to that company is reported in C. I. O. S. File XXXIII-17, Item 30 (PB 28,744), "Coal Driers, Büttner-Werke A. G., Uerdingen-Krefeld", 1945, by H. Bardgett, which also give further information concerning Büttner driers.

The Blümner Process: Frames 23-26; 54-47.

About 1936, Dr. E. Blümner developed a process in Germany for the low-temperature carbonization of coal. The Borsig Company investigated this process but did not carry it beyond the project stage. Dr. Blümner then organized the Blümner Fuel Corp. in London but was unable to obtain sufficient backing for his enterprise. During the war, experts of the German

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Government became interested in the process and obtained Government aid for the construction of a semicommercial plant with a capacity of 10 metric tons of bituminous coal per day. A site for the plant was selected in Berlin but had to be abandoned because of air raids. Another site was then chosen near Munich, and construction was started, but the plant had not been completed when the war ended.

Figure 13 (frame 54) shows the flow sheet for a Blümner process plant having a daily capacity of 100 metric tons of bituminous coal. According to this flow sheet, 60 metric tons of powdered coal is mixed with 40 metric tons of oil. The mixture is then subjected to a pressure of 30 atmospheres (441 pounds per square inch), preheated, emulsified, extracted, distilled, and decomposed in six steps. Distillate oil and a residue consisting of a mixture of coal and oil are the two products resulting from these six operations.

From the distillate oil are obtained 40 metric tons of oil to be returned to the process for mixing with more coal, 6.5 metric tons of fuel oil, 6.5 metric tons of Diesel oil, and 8.5 metric tons of crude benzine. The residual mixture of coal and oil is mixed with 40 metric tons of fine coal, and this mixture is carbonized to yield 65 metric tons of coke. The gas generated in the process is used for heating purposes.

Figure 14 (frame 55) shows the arrangement planned for a Blümner plant. The various parts of the plant are as follows: (1) Delivery of powdered coal, (2) coal-dust bin, (3) oil-storage tank, (4) high-level tank for oil, (5) coal-feeding arrangement, (6) oil-measuring arrangement, (7) mixer for coal and oil, (8) pressure pump for colloidal fuel (coal-oil mixture), (9) storage tank for colloidal fuel, (10) operating tank for colloidal fuel, (11) pressure pump for hot colloidal fuel, (12) preheater, (13) retorts with hollow revolving cylinders inside, (14) oven setting, (15) vapor off-take, (16) expansion vessel, (17) drain pipe, (18) carbonizing ovens, (19) return pipe for colloidal fuel, (20) furnace for expansion vessel, (21) vapor off-take, (22) rectifying column, (23) dephlegmator, (24) gas outlet, (25) water cooler, (26) separator, (27) light-oil overflow, (28) water off-take, (29) collecting main, (30) hot-oil pipe, (31) oil cooler, (32) pipe for gases and vapors, (33) Diesel oil-drain pipe, (34) Diesel-oil cooler, (35) Diesel-oil off-take, (36) fuel-oil pipe, (37) fuel-oil cooler, and (38) fuel-oil off-take.

According to Dr. Thau, "The most vital parts of this process are the cylindrical steel retorts in which the colloidal fuel is heated and agitated under a pressure of about 30 atmospheres (441 pounds per square inch)." The arrangement of one of these retorts is shown in vertical cross section in figure 15 (frame 56) and in horizontal cross section in figure 16 (frame 57).

Dr. Thau comments as follows on the future of the Blümner Process: "This is no doubt a very interesting process, but a definite judgment as to its success cannot be passed until the results of the trial plant, still under construction (near Munich), are available."

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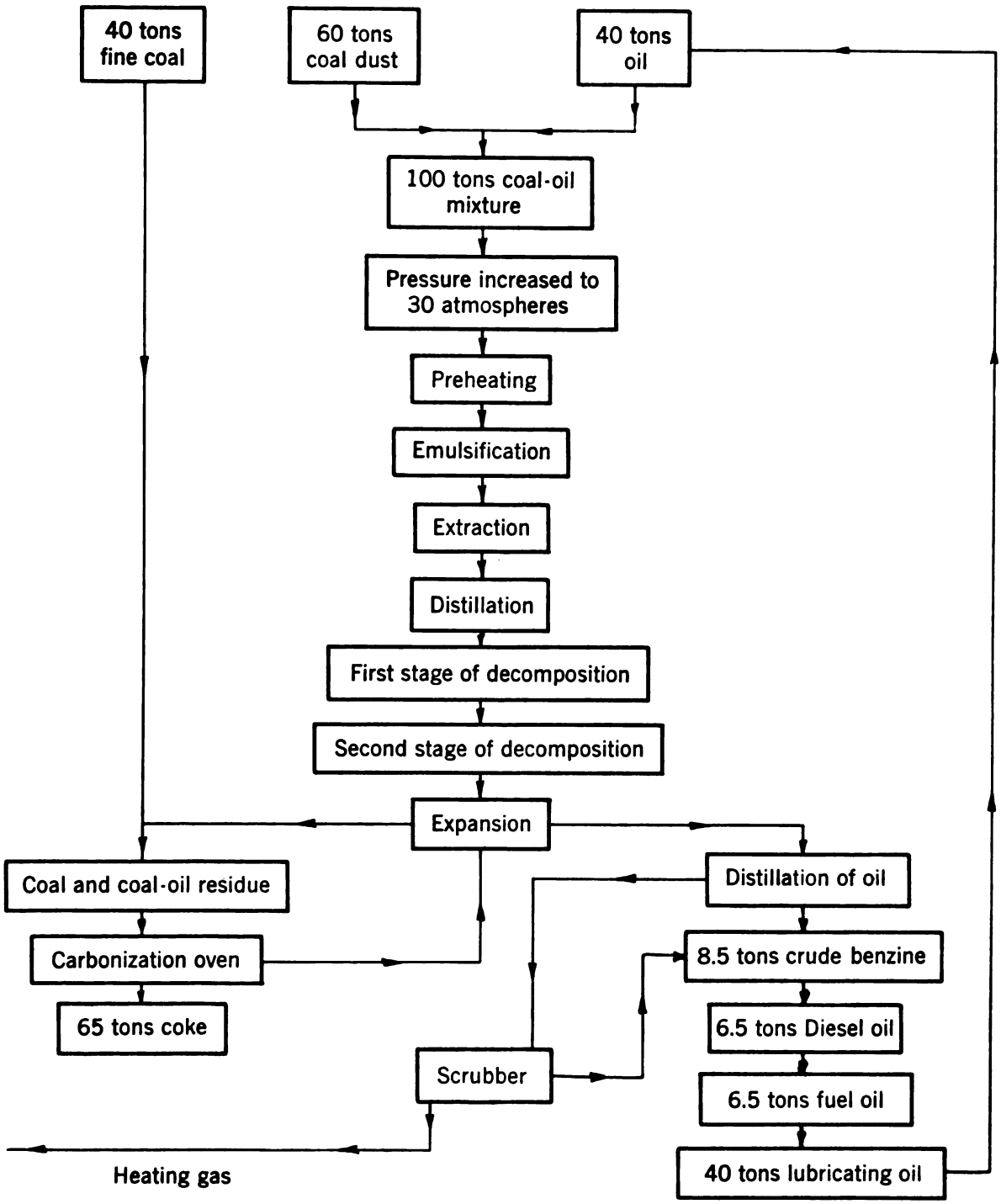

Figure 13. - Flow sheet for Blümner process (throughput, 100-metric tons of coal daily).

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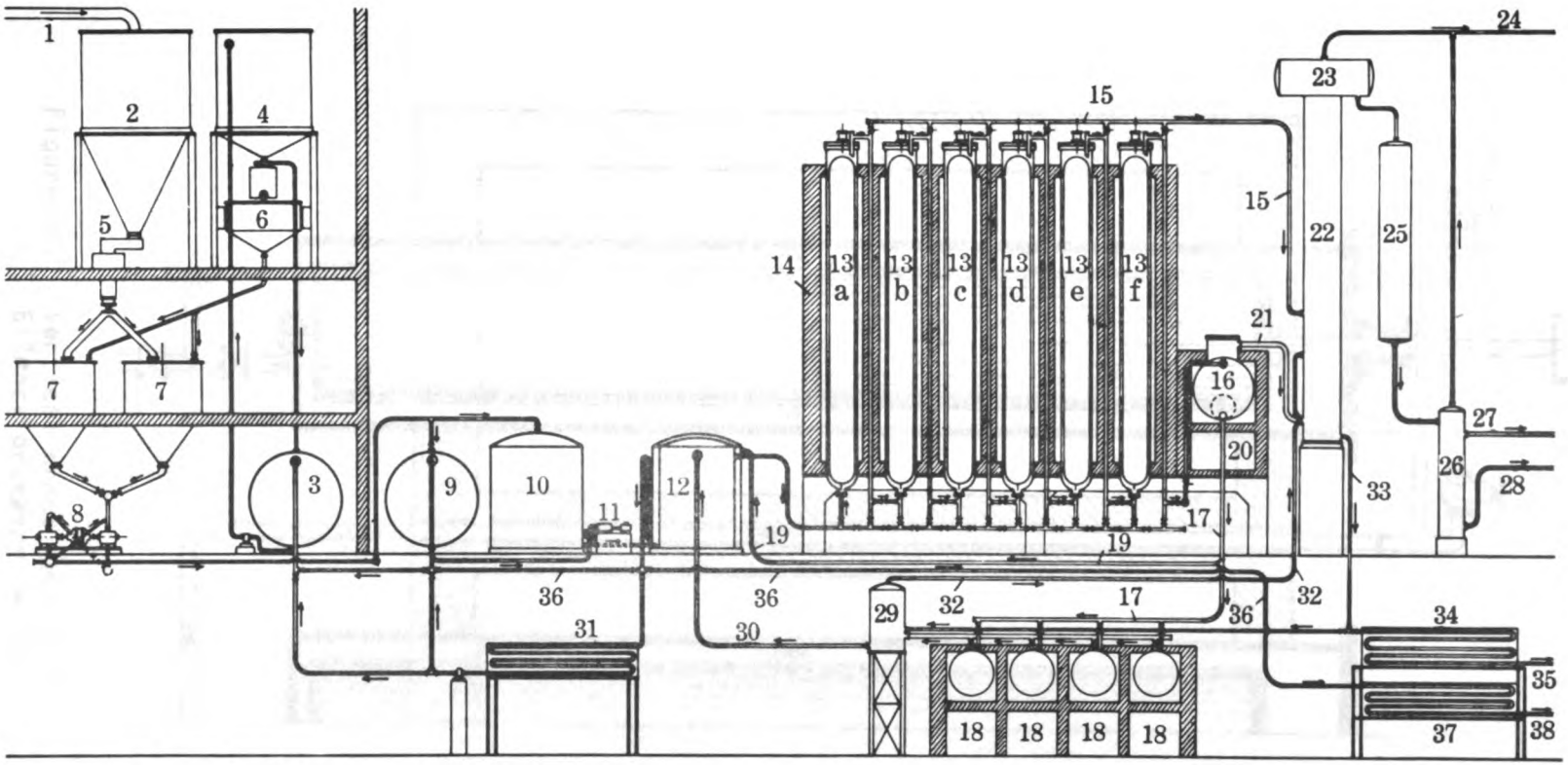

Figure 14. - Arrangement of equipment in Blümner plant (see text for numbering of parts).

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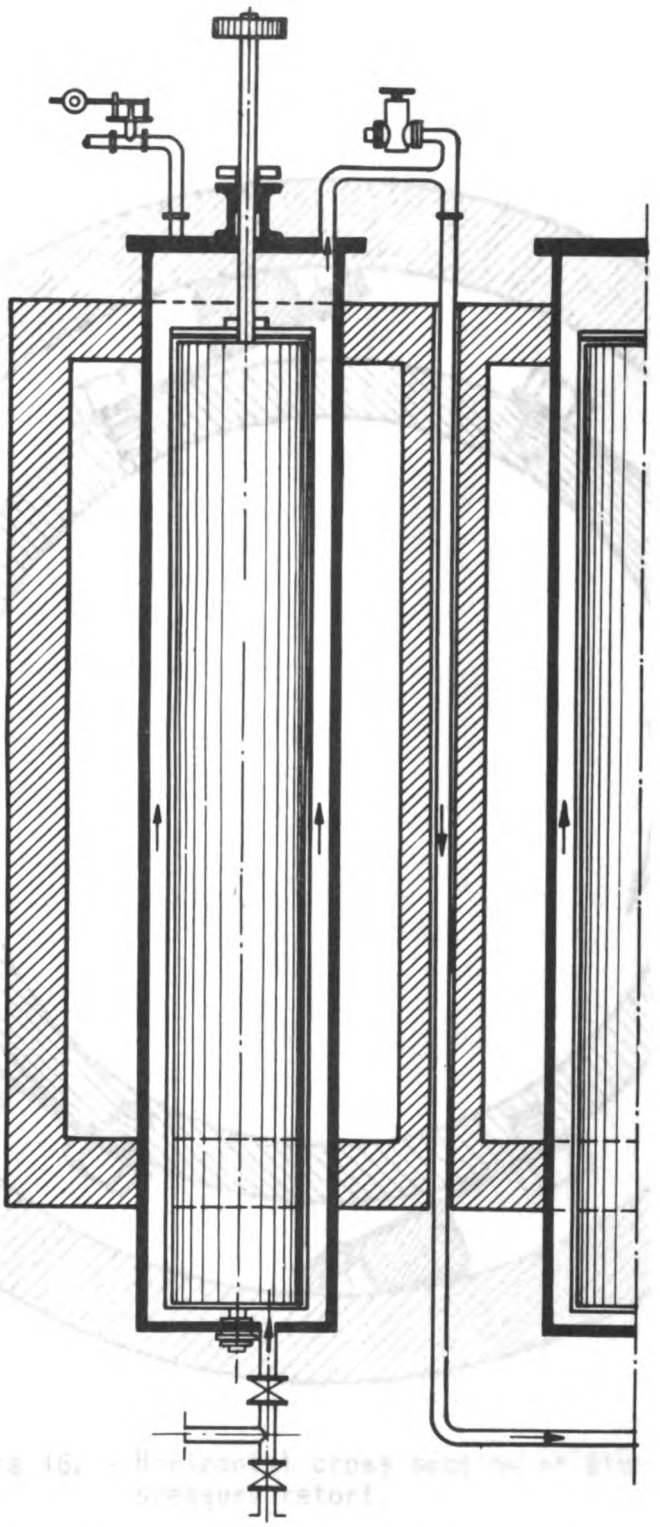

Figure 15. - Vertical cross section of Blömner pressure retort.

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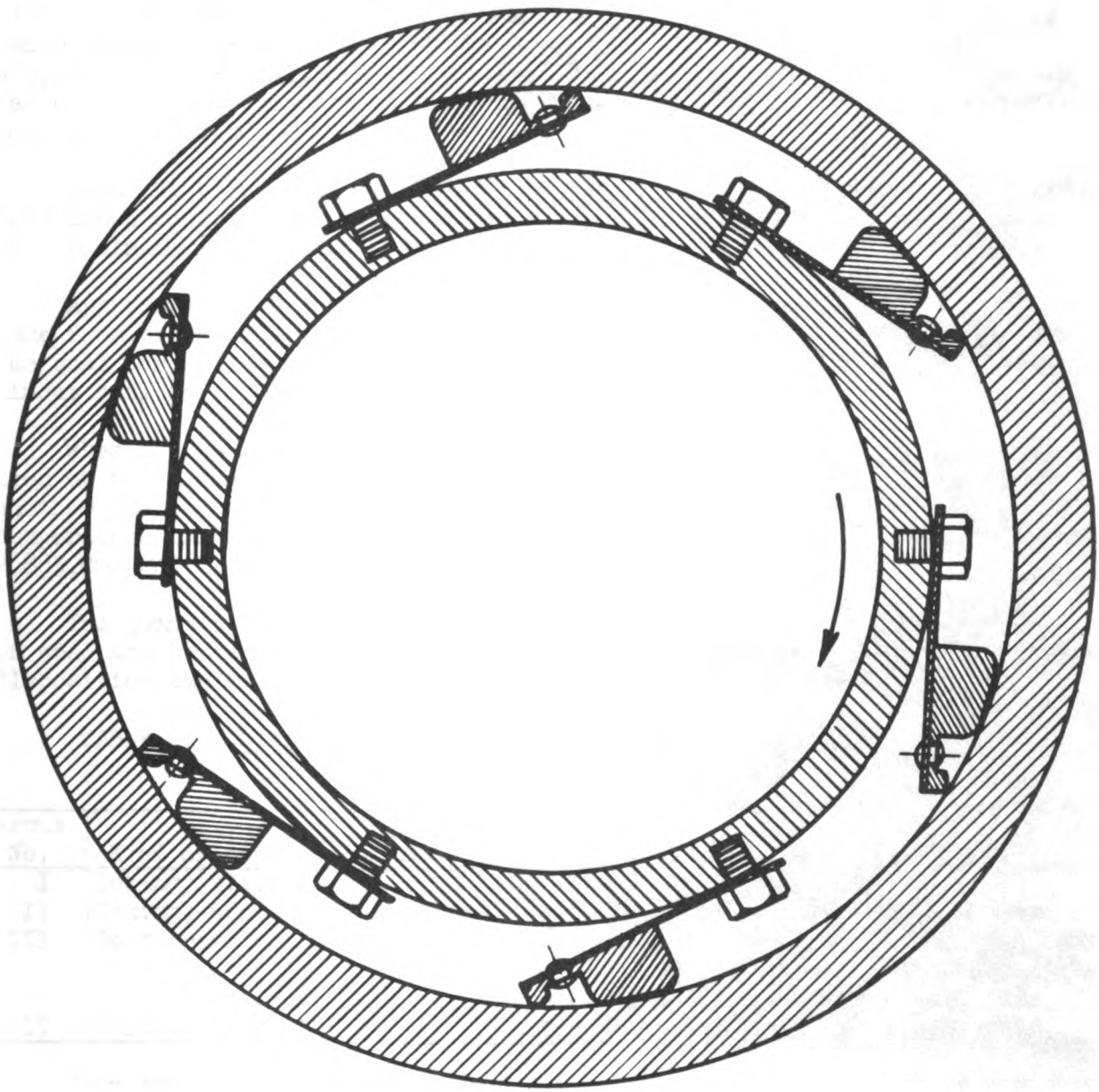

Figure 16. - Horizontal cross section of Blümner pressure retort.

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Quantities of Low-Temperature Tar Produced and Their Uses in 1943

The quantity of low-temperature tar produced in 1943 by plants owned or operated by German interests was approximately 2,633,000 metric tons or 658,250,000 U. S. gallons. It is interesting to note that the total quantity of high-temperature tar processed in that same year by all tar plants under German control was approximately 2,289,000 metric tons or about 503,580,000 U. S. gallons. In other words, the quantity of low-temperature tar exceeded the quantity of high-temperature tar by approximately 344,000 tons or 154,670,000 gallons.

About 91 percent of the total low-temperature tar produced in 1943 came from brown coal. Of this, 84 percent came from Lurgi-Spülgas retorts and the remainder, 7 percent, from Geissen, Borsig-Geissen and Rolle retorts.

Approximately 9 percent of the low-temperature tar produced in 1943 came from bituminous coal. Of this, 8 percent came from noncaking bituminous coal carbonized in Lurgi-Spülgas retorts and 1 percent from caking bituminous coal carbonized by the Krupp-Lurgi process.

When the war ended, four new plants for the low-temperature carbonization of bituminous coal were under construction. They would have produced another quarter of a million metric tons or 62,500,000 U. S. gallons of low-temperature tar if they had been completed, making the total production about 2,883,000 metric tons or 720,750,000 U. S. gallons per year.

The quantities of low-temperature tar produced in 1943 from each kind of coal and by each plant were given in table 1, page 12. Table 2 shows what disposition was made of the low-temperature tar from each kind of coal.

TABLE 2. - Disposition of low-temperature tar from each kind of coal carbonized in 1943

Group No.	Kind of coal carbonized	Tons of tar (metric)	Disposition of tar
I	Coking, bituminous	28,000	Navy fuel.
II	Caking, noncoking bituminous	205,000	Hydrogenation to fuel oils.
III	Soft brown coal	600,000	Distillation to fuel oils, paraffin wax, and electrode coke.
	do.	800,000	Hydrogenation to fuel oils.
IV	Hard brown coal	1,000,000	Hydrogenation to fuel oils.

From the above it may be observed that hydrogenation to fuel oils was the outlet for 2,005,000 metric tons of low-temperature tar or 76.1 percent of the total amount produced. Six-hundred thousand metric tons, or 22.8 percent, was distilled and processed for the recovery of fuel oils, paraffin wax, and electrode coke. Twenty-eight thousand metric tons, or about 1.1 percent, was used directly as fuel oil by the Navy. From this it may be seen that substantially all of the low-temperature tar was used for fuel purposes. Virtually the only other products were paraffin wax and electrode coke. These products were made from the 600,000 metric tons of low-temperature tar which were distilled.

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Processing of Low-Temperature Tar

In general, it may be said that three methods were used for the processing of the low-temperature tar produced in Germany during World War II: (1) Blending with oils from other sources, (2) hydrogenation, and (3) distillation and cracking.

Blending With Oils From Other Sources: Frames 599-606.

Much of the low-temperature tar produced from coking bituminous coal in the Krupp-Lurgi installations at Wanne-Eickel (Ruhr) and Velsen (Saar) was blended with high-temperature tar oils and aromatic fuel oils from Fischer-Tropsch synthetic-fuel plants or from Bergius hydrogenation plants.

Some mixtures of low-temperature tar and paraffinic oils were prepared. According to Dr. Demann of Fried. Krupp A.G., it was found that the addition of phenols or phenylhydrazine to the low-temperature tar would prevent sludging of such mixtures, but to avoid the use of admixtures attempts were made to remove the asphaltenes from the tar before adding it to the paraffinic oil. The procedure developed for this purpose consisted in diluting the low-temperature tar with benzene in the proportion of ten parts benzene to one part tar. The asphaltenes precipitated by the benzol were separated from the liquid portion of the benzene-tar mixture by decantation, and the latter was then extracted with dilute caustic soda to remove phenols and distilled under vacuum to remove the benzene. The yield of asphalt-free low-temperature tar extract made in this manner was said to be 80 percent, the remainder being asphalt and phenols. According to Dr. Demann, the oil freed of asphalt and tar acids in this manner may be used to make additional low-temperature tar miscible with paraffinic oils.

An interesting method of measuring the miscibility of aromatic and paraffinic oils by means of a Höppler viscosimeter was described by Dr. Demann. Mixtures of the two materials under investigation are made in different proportions. The viscosity of each mixture is determined with two balls of different sizes. Mixtures that show no tendency to sludge give the same viscosity reading with either ball, but the two determinations are different when there is actual or incipient sludging. By this means, ranges of miscibility and nonmiscibility may be established for mixtures of aromatic and nonaromatic oils.

Hydrogenation

As stated previously in this report, "hydrogenation to fuel oils was the outlet for 2,005,000 metric tons of low-temperature tar or 76.1 percent of the total amount produced." From this it may be seen that hydrogenation was, by far, the most important method of processing to which low-temperature tar was subjected.

The conversion of low-temperature tar or combinations of brown coal and low-temperature tar to liquid fuels by hydrogenation has been described in detail by other investigators. Complete descriptions of such processes are

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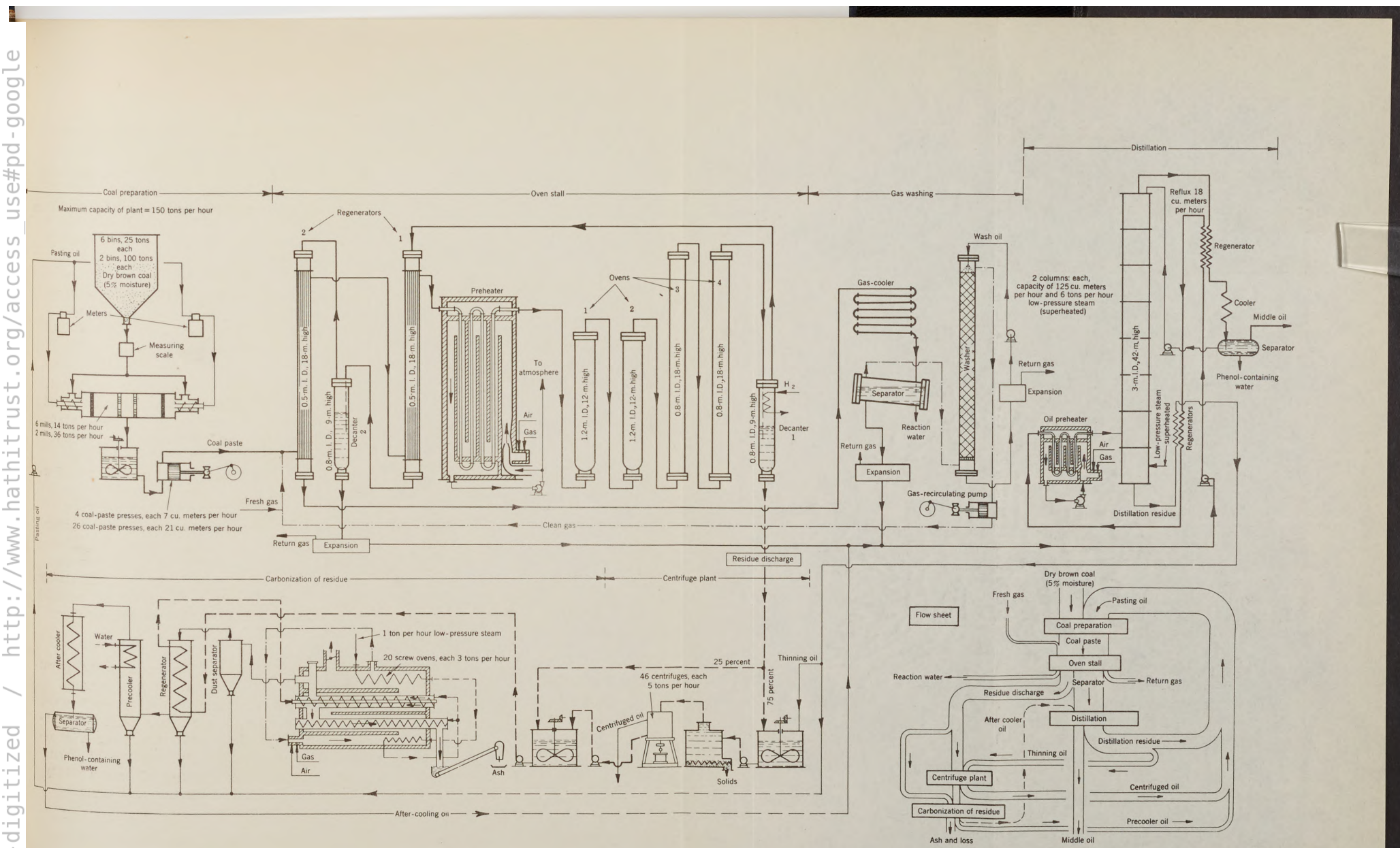

Figure 17. - Flow diagram of coal-hydrogenation plant (prior to 1937) at Leuna, I. G. Farbenindustrie, Merseburg.

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given in Bureau of Mines Information Circular 7370, July 1946, "Report on the Investigation by Fuels and Lubricants Teams of the I. G. Farbenindustrie A. G., Leuna Works, Merseburg, Germany", edited by R. Holroyd. This comprehensive compiliation is also issued as C. I. O. S. File XXXII-107, Item 30 (PB 6,650), "Investigation by Fuels and Lubricants Team at the I. G. Farbenindustrie A. G. Works at Leuna", 1945, by R. Holroyd, Editor. Flow diagrams are given for the conversion at Leuna of raw brown-coal tar to middle oil and the conversion of the middle oil either to motor gasoline or to aviation-base gasoline. Flow diagrams also are given for the hydrogenation of mixtures of raw brown-coal tar and brown coal to produce motor gasoline. Information is given in the same report converning the hydrogenation of bituminous coal at Blechhammer and hard brown coal at Most (Brüx). Figure 17 (frame 673) illustrates the brown-coal hydrogenation process as carried out at Leuna.

Distillation and Cracking

The 600,000 metric tons of low-temperature tar distilled in Germany in 1943 were processed in five plants. Four of these plants, handling a total of about 400,000 metric tons per year, used similar methods of distillation and paraffin-wax recovery. The names and locations of these plants are as follows: Riebecksche Montanwerke, Webau; Anhalt. Kohlenwerke, Köpsen; Deutsche Erdöl A. G., Rositz; and K. V. G., Gölzau.

The fifth plant, belonging to Aktiengesellschaft Sächsische Werke, was located at Espenhain near Leipzig. It handled about 200,000 metric tons of tar per year. It employed tar-distilling, oil-redistilling, and pitch-carbonizing equipment installed during the war by the Carl Still organization of Recklinghausen and a solvent-dewaxing process developed by Edeleanu Gesellschaft m.b.H., Berlin. The latter process was first tried in a pilot plant by Deutsche Erdöl A. G. at Rositz.

Following are brief descriptions of the methods employed for the processing of low-temperature brown-coal tar by Deutsche Erdöl A. G. at Rositz and by Akt. Sächsische Werke at Espenhain. They are based upon information received from Deutsche Erdöl (frames 755-764); Edeleanu Ges. (frames 739-754), and Carl Still (frames 636-658).

Deutsche Erdöl A.G. Plant at Rositz: Frames 755-764. - Two processes were used for working up the low-temperature brown-coal tar at Rositz. One was a distillation process and was operated primarily to obtain paraffin wax and heating oil. The purpose of the other process, which employed cracking, was to obtain Diesel motor oil and heating oil.

The distillation process involved the following steps:

1. Distillation of the low-temperature tar to obtain crude benzine, heating oil, crude-paraffin oil, and asphalt.

2. Redistillation of the crude paraffin oil to recover additional heating oil and asphalt and to make the paraffin oil more readily crystallizable.

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3. Cooling of the redistilled paraffin oil to 18° C. (64.4° F.) and filtering to obtain hard-paraffin wax and a filtrate oil containing soft-paraffin wax.

4. Cooling to -2° C. (28.4° F.) and filtration of the above filtrate to obtain soft-paraffin wax and a filtrate of heating-oil quality. The soft wax goes to paraffin oil II.

5. The hard-paraffin wax is washed with caustic soda and water to remove phenols. The phenols are liberated from the caustic solution with carbon dioxide and added to the heating oil.

6. The washed, hard-paraffin wax is separated by sweating into forerunnings high in oil and of low-paraffin content, a residue reasonably oil-free but high in paraffin, and oil-free paraffin. The forerunnings go to paraffin oil II, and the residue, by repeated sweatings, is separated into oil-free paraffin and paraffin oil II.

7. The sweated paraffin is refined with sulfuric acid. The refined paraffin is separated from solids by centrifuging and then cast in blocks.

8. The sulfuric sludge containing some paraffin is extracted with benzine and separated from insoluble matter by filtration. The benzine solution is distilled to recover the benzine and to obtain paraffin, which is returned to step 5.

9. Asphalt obtained from the initial distillation of the tar and from the redistillation of the crude-paraffin oil is distilled to electrode coke. The distillate containing paraffin is added to paraffin oil II.

10. Paraffin oil II, consisting of soft-paraffin wax from steps 4 and 6 and asphalt distillate from step 9 is redistilled to obtain redistilled paraffin oil II, heating oil, and asphalt. The redistilled paraffin oil II is then treated in the manner described in steps 3 to 9.

The following products were made at Rositz by this process in 1942-1943 from 150,000 metric tons of crude low-temperature, brown-coal tar:

	Metric tons	Percent
Heating oil	97,500	65.0
Paraffin	19,500	13.0
Electrode coke	12,750	8.5
Fuel coke	3,750	2.5
Gas and loss	16,500	11.0

The cracking process used at Rositz for working up brown-coal tar was operated as follows:

1. The low-temperature tar was topped to 300° C. (572° F.) to remove crude benzine and heating oil as distillates and to improve the quality of the products from the cracking operation.

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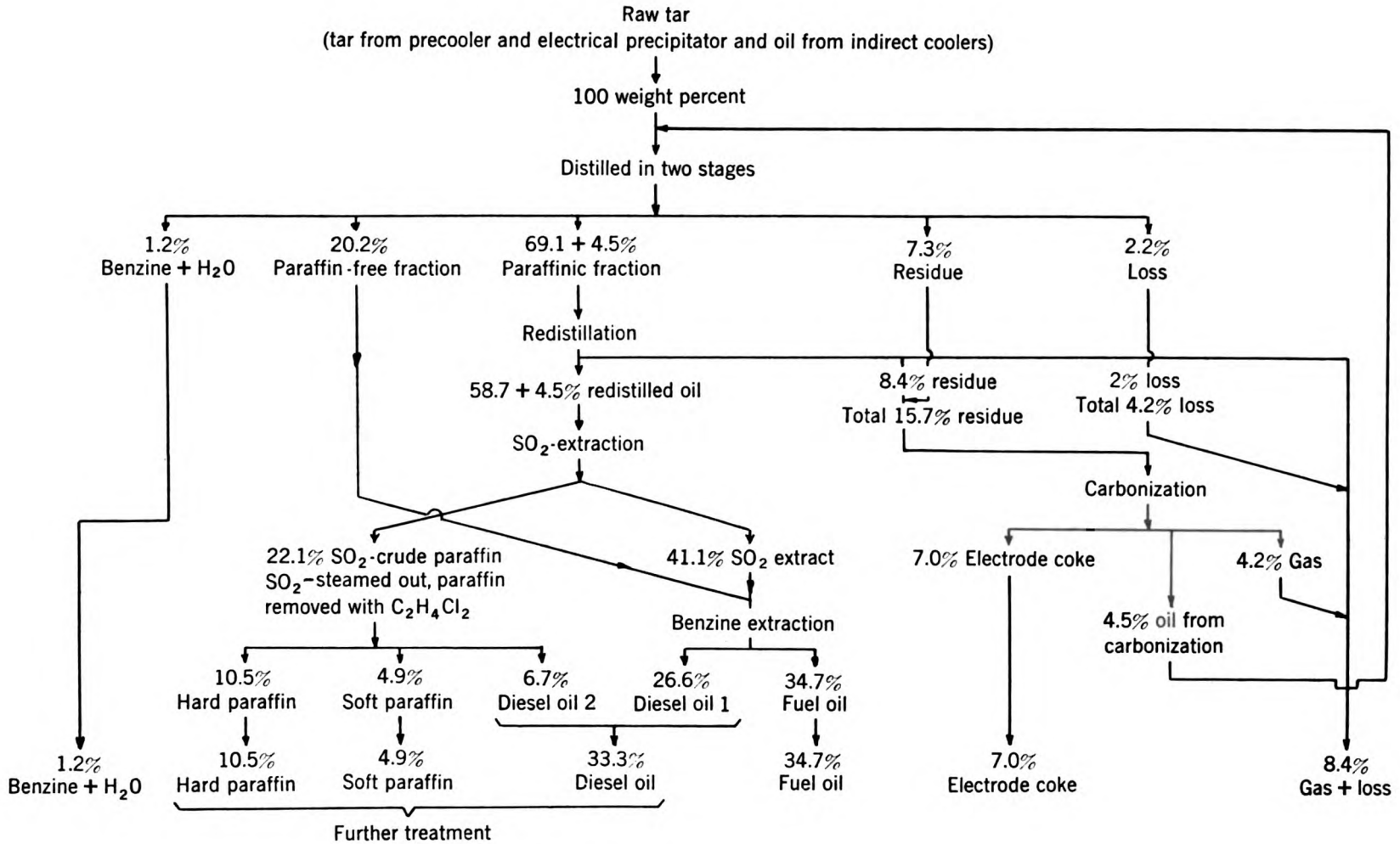

Figure 18. - Edeleanu process for refining brown-coal tar by distillation and by treatment with selective solvents.

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2. By pressure treatment at 430°-450° C. (806°-842° F.) and 40-60 atmospheres (588-882 pounds per square inch), the topped tar was cracked to obtain crude-pressure benzine, pressure gas oil, asphalt, and cracking still gas. The latter was used for heating purposes.

3. The pressure benzine was refined with caustic soda and sulfuric acid and separated by fractionation into benzine and gas oil, whose subsequent treatments gave finished products.

4. The crude pressure-gas oil was refined with caustic soda and sulfuric acid and redistilled. The distillate, after treatment with caustic soda and Fuller's earth, was gas oil or Diesel oil.

5. The refining and distillation residues were distilled, together with the cracked asphalt, to a solid residue, which was graded according to its asphalt content as fuel coke or electrode coke. The asphalt distillate was returned to the other process for paraffin recovery.

The products made at Rositz by this cracking process in 1943-1943 from 80,000 metric tons of tar were as follows:

	Metric tons	Percent
Heating oil	37,600	47.0
Benzine, 150° C.	2,400	3.0
Benzine, 150°-185° C. ..	800	1.0
Diesel oil	16,000	20.0
Electrode coke	5,600	7.0
Fuel coke	2,400	3.0
Brown-coal-tar pitch ...	4,800	6.0
Gas and loss	10,400	13.0

A. G. Sächsische Werke Plant at Espenhain: Frames 636-658; 739-754. - In the years immediately preceding and during the early part of World War II, a large plant was erected at Espenhain, Germany, by A. G. Sächsische Werke under the auspices of the German Reich. It included a brown-coal mine; electric-power plant; a Lurgi Spülgas plant for the low-temperature carbonization of the brown coal to produce low-temperature coke for the power plant, low-temperature tar, and light oil; a tar-distilling, oil-redistillation, and pitch-coke plant; and an Edeleanu plant for the solvent refining of the tar distillates to recover paraffin wax, heating oil, and Diesel oil. A description of the plant is given in C. I. O. S. File XXVIII-23, Item 30 (PB 1,106), "A. G. Sächsische Werke-Espenhain, Kreis Borna, Germany", 1945, by G. S. Bays, J. P. Jones, and B. L. Mackusick; also available as a part of TOM Microfilm Reel 196.

The manner in which the Edeleanu process is employed in connection with the refining of brown-coal tar is illustrated by figure 18 (frame 752).

The following information concerning the tar-distillation, pitch-carbonizing, and Edeleanu sections of the plant is based upon reports received from the Carl Still and Edeleanu organizations.

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Tar distillation. - The operation of the tar-distilling section of the plant is illustrated by the flow diagrams in figures 19 and 20 (frames 640 and 641). A brief description of each operation follows.

Tar centrifuging: Frames 643-645.

The crude brown-coal tar, together with distillate oil from the pitch-coke plant, is centrifuged to remove entrained solids and water (fig. 21, frame 645). The tar is pumped to an elevated feed tank, where it is heated by steam coils. It then flows by gravity to horizontal centrifuges. Excess tar, which does not enter the centrifuges, flows to a collecting tank, from which it is pumped back to the feed tank. The tar enters the centrifuges through charging valves, which are operated by air or oil pressure. When the basket of a centrifuge is filled, the valve automatically closes. The tar in the basket of the centrifuge separates into three layers; namely, a solid residue, a watery intermediate layer, and clean tar. The tar is removed first by means of a tube directed by hand. The intermediate layer is removed next in the same manner, and later is centrifuged again to separate the tar and water. The residue is then removed by a plow operated by oil or air pressure. The residue slides down a chute into a truck, by which it is transferred to cars. The casings of the centrifuges are heated. Vapors from the centrifuges are conveyed to coolers. The centrifuged tar goes to tanks, from which it is pumped to the atmospheric tar still.

Atmospheric distillation: Frames 646-649.

The centrifuged crude tar is pumped through a series of heat exchangers and condensers to a gas-heated pipe still (fig. 22, frame 649), from which it discharges into a combined flash chamber and fractionating column operating under atmospheric pressure. Steam superheated in the convection section of the pipe still is introduced into the bottom of the flash chamber. Light oil and water leave the column at the top. Paraffin-free and paraffin-containing fractions leave the column as side streams. The residue from this atmospheric distillation leaves the bottom of the column and is pumped to the vacuum still.

Vacuum distillation: Frames 646-649.

The residue from the atmospheric still is pumped through a second pipe heater (fig. 22, frame 649), from which it is passed through a valve into a combined flash chamber and a column operating under about 50 mm. (about 2 inches) Hg vacuum absolute. Superheated steam is introduced into the bottom of the flash chamber, water and a distillate containing paraffin leave the top of the column, and a second distillate containing paraffin is taken as a side stream from the column. The residue from this operation is soft pitch.

Distillation to hard pitch: Frames 456-458, FIAT Microfilm Reel C-96 or TOM Microfilm 150, (PB L 13,096).

The soft pitch from the vacuum still is transferred to either of two intermittent, gas-heated stills operating under vacuum. Figure 23 (frame

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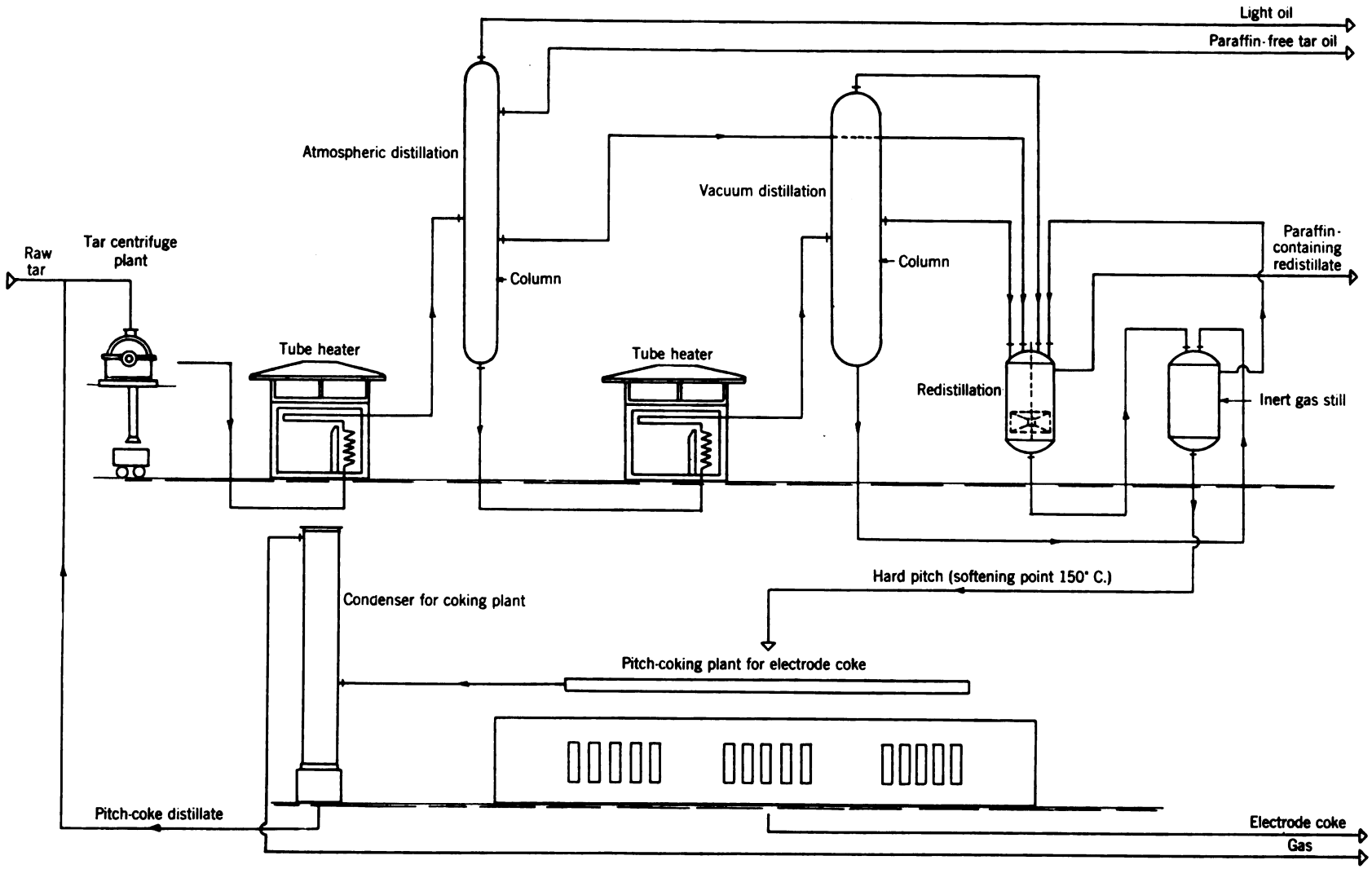

Figure 19. - Arrangement for tar distillation, A. G. Sächsische Werke, Espenhain.

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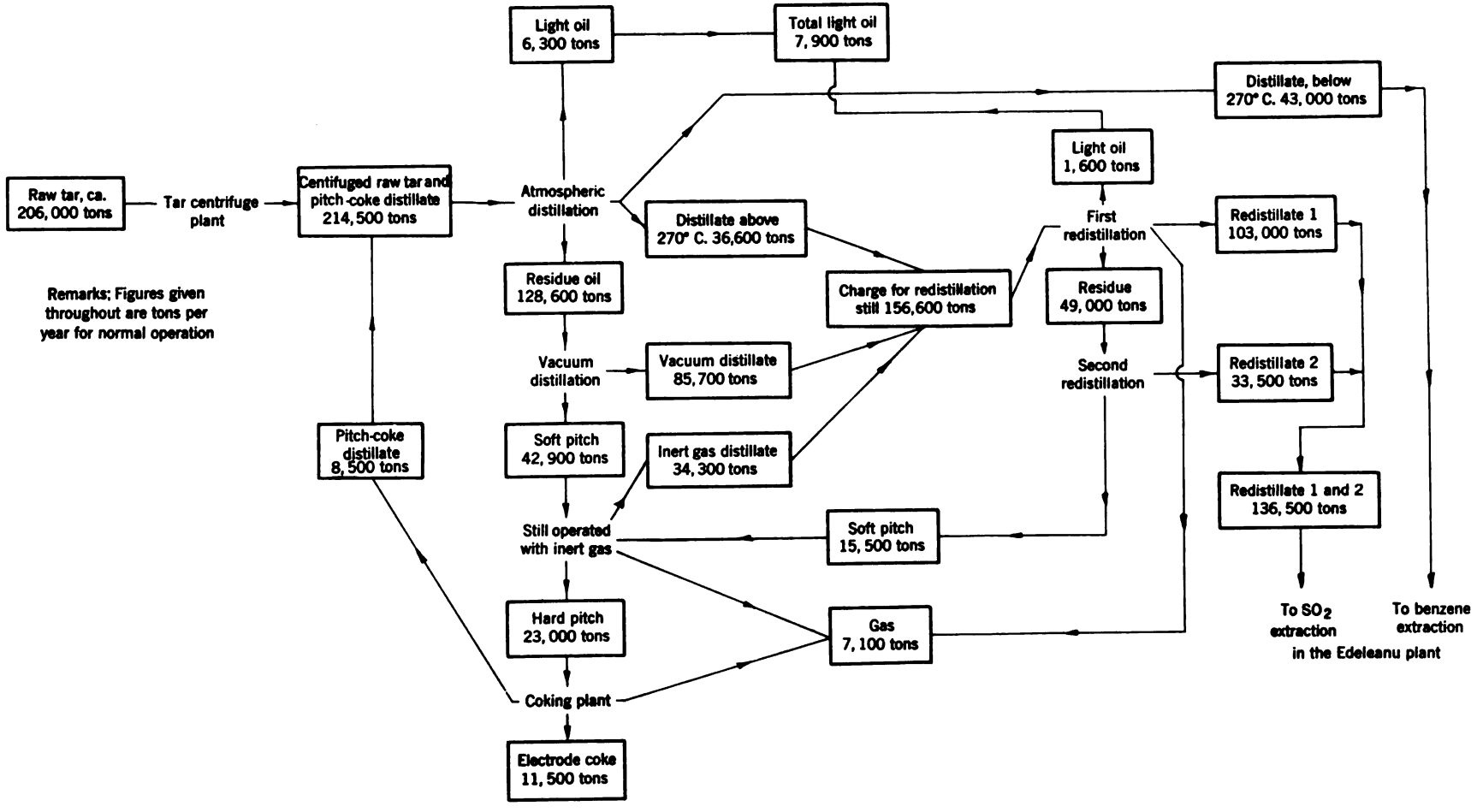
Raw tar, ca. 206,000 tons
Tar centrifuge plant
Centifuged raw tar and pitch-coke distillate 214,500 tons
Pitch-coke distillate 8,500 tons
Remarks; Figures given throughout are tons per year for normal operation
Light oil 6,300 tons
Total light oil 7,900 tons
Atmospheric distillation
Distillate above 270° C. 36,600 tons
Residue oil 128,600 tons
Vacuum distillation
Vacuum distillate 85,700 tons
Soft pitch 42,900 tons
Inert gas distillate 34,300 tons
Still operated with inert gas
Hard pitch 23,000 tons
Coking plant
Electrode coke 11,500 tons
Distillate, below 270° C. 43,000 tons
Light oil 1,600 tons
First redistillation
Charge for redistillation still 156,600 tons
Residue 49,000 tons
Second redistillation
Soft pitch 15,500 tons
Gas 7,100 tons
Redistillate 1 103,000 tons
Redistillate 2 33,500 tons
Redistillate 1 and 2 136,500 tons
To SO_2 extraction in the Edeleanu plant
To benzene extraction

Figure 20. - Flow diagram for tar distillation, A. G. Sächsische Werke, Espenhain.

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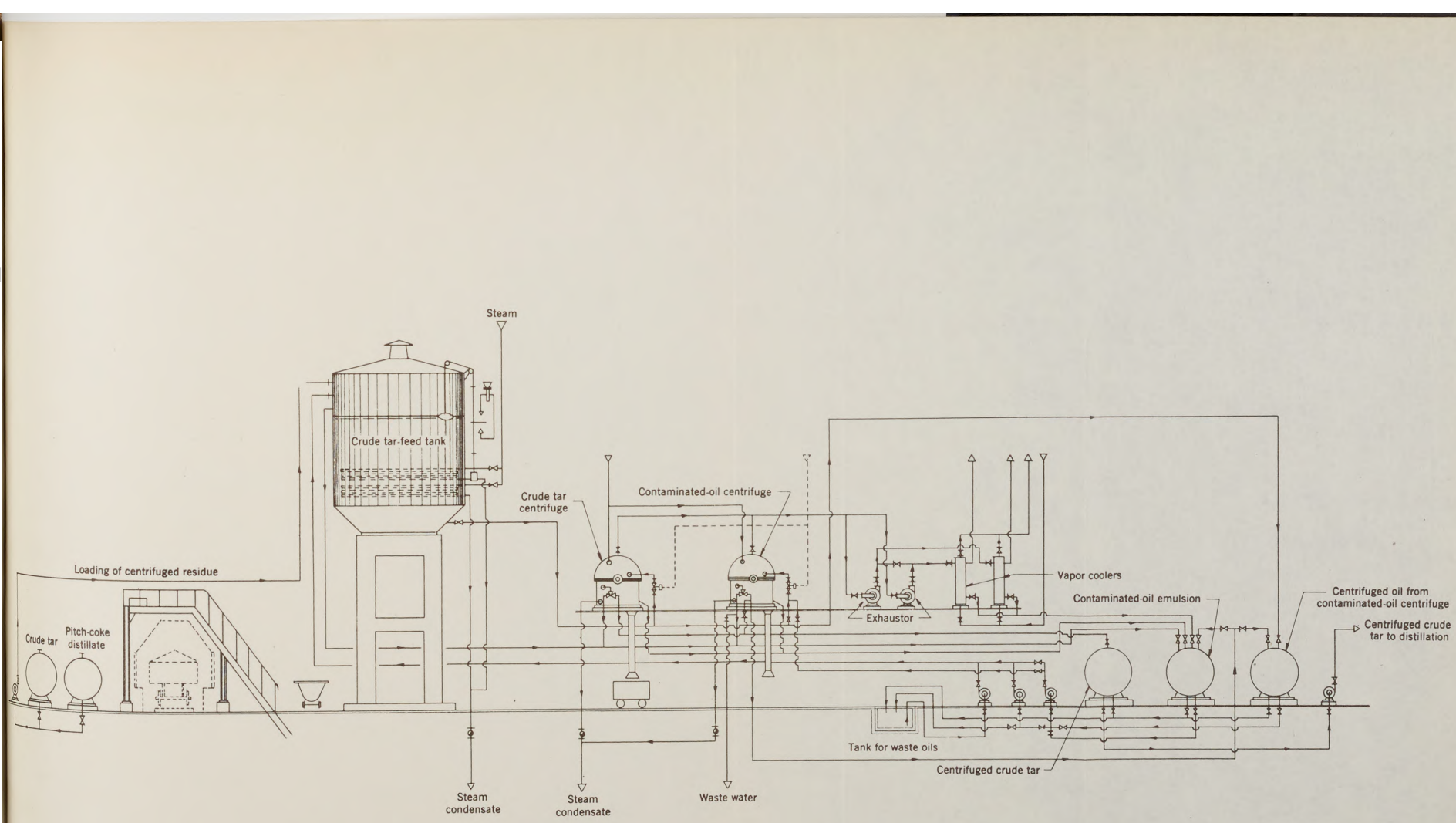

Figure 21. - Flow diagram for crude, brown-coal-tar centrifuge plant, A. G. Sächsische Werke, Espenhain.

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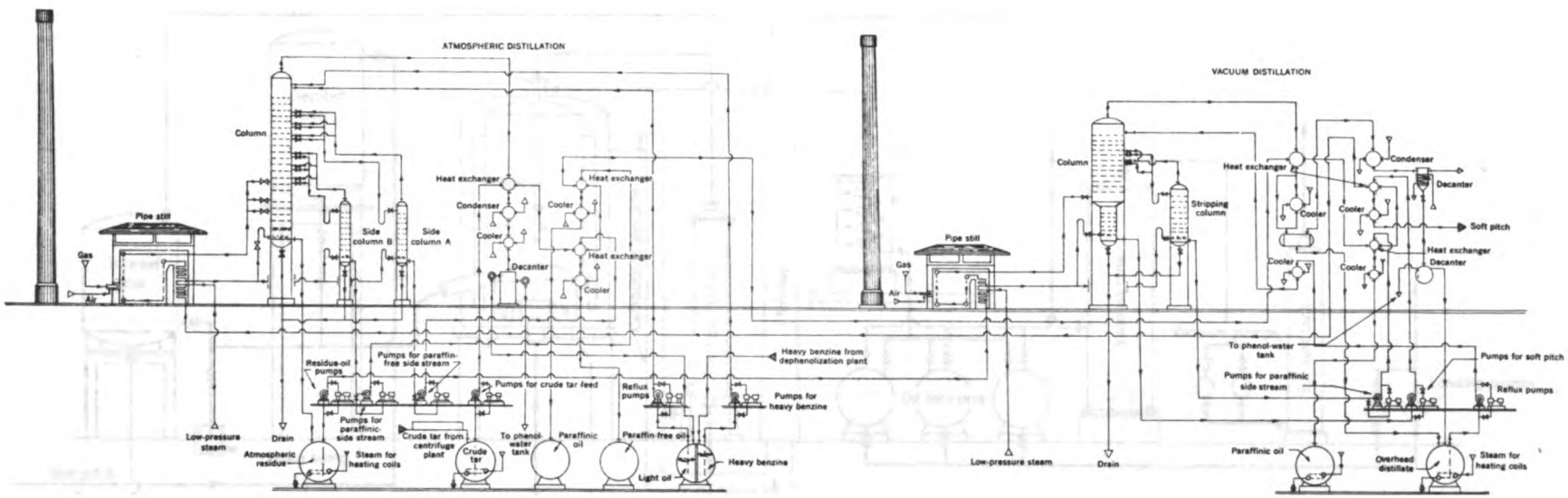

Figure 22. - Flow diagram for atmospheric and vacuum distillation of tar, A. G. Sächsische Werke, Espenhain.

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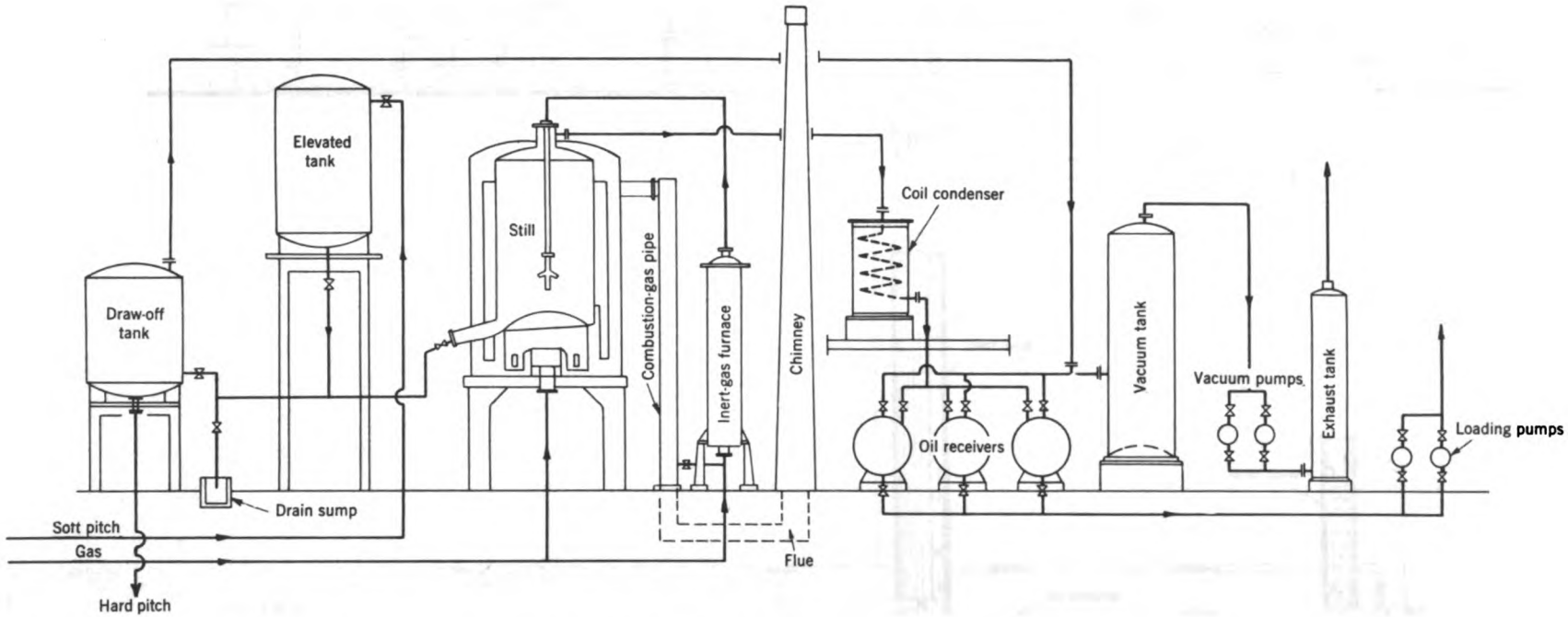

Figure 23. - Plant for producing hard pitch (softening point, 150° C. or 302° F.) by direct heating with inert gas, A. G. Sächsische Werke, Espenhain.

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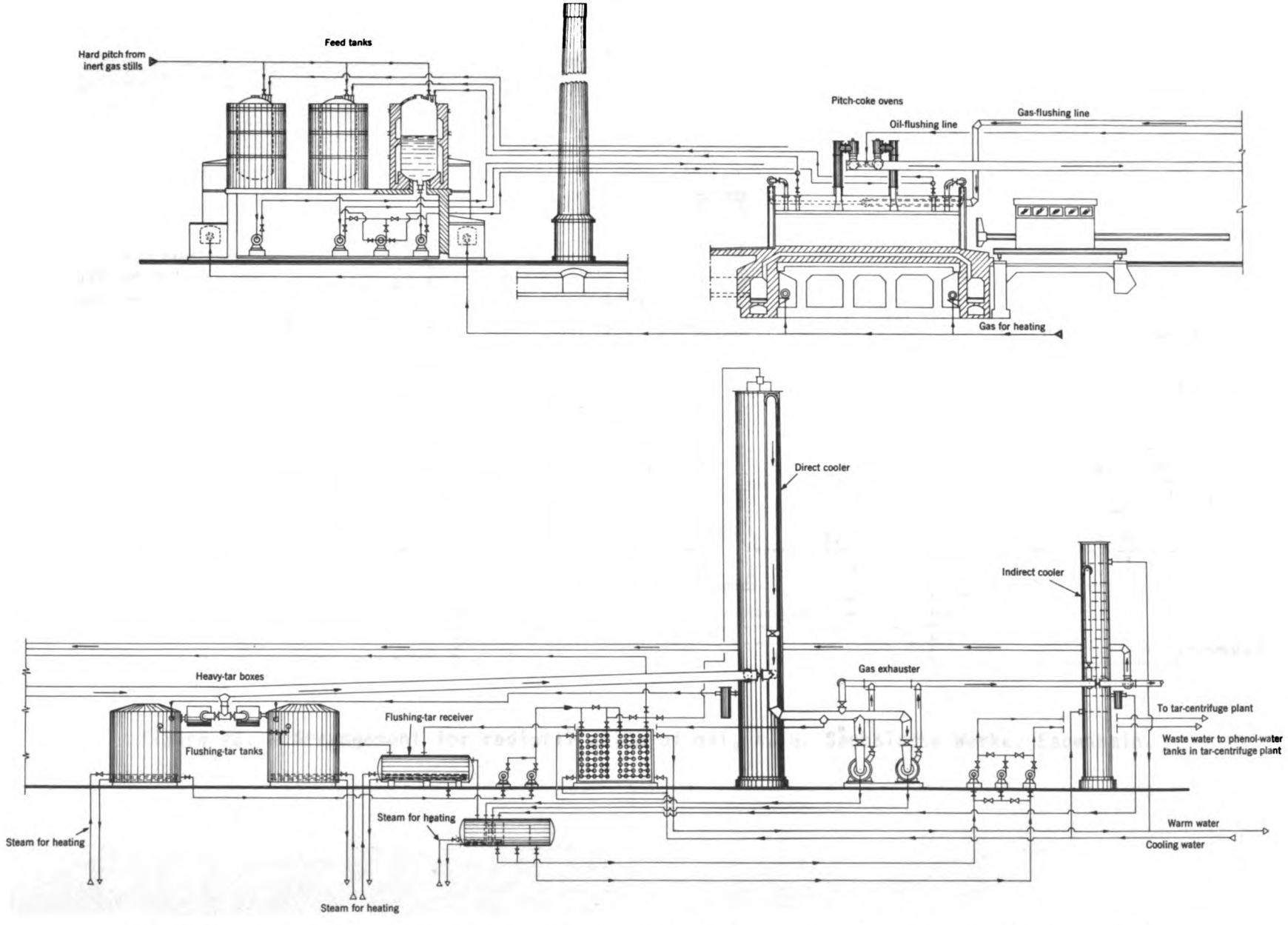

Figure 24. - Arrangement of pitch-carbonization plant, A. G. Sächsische Werke, Espenhain.

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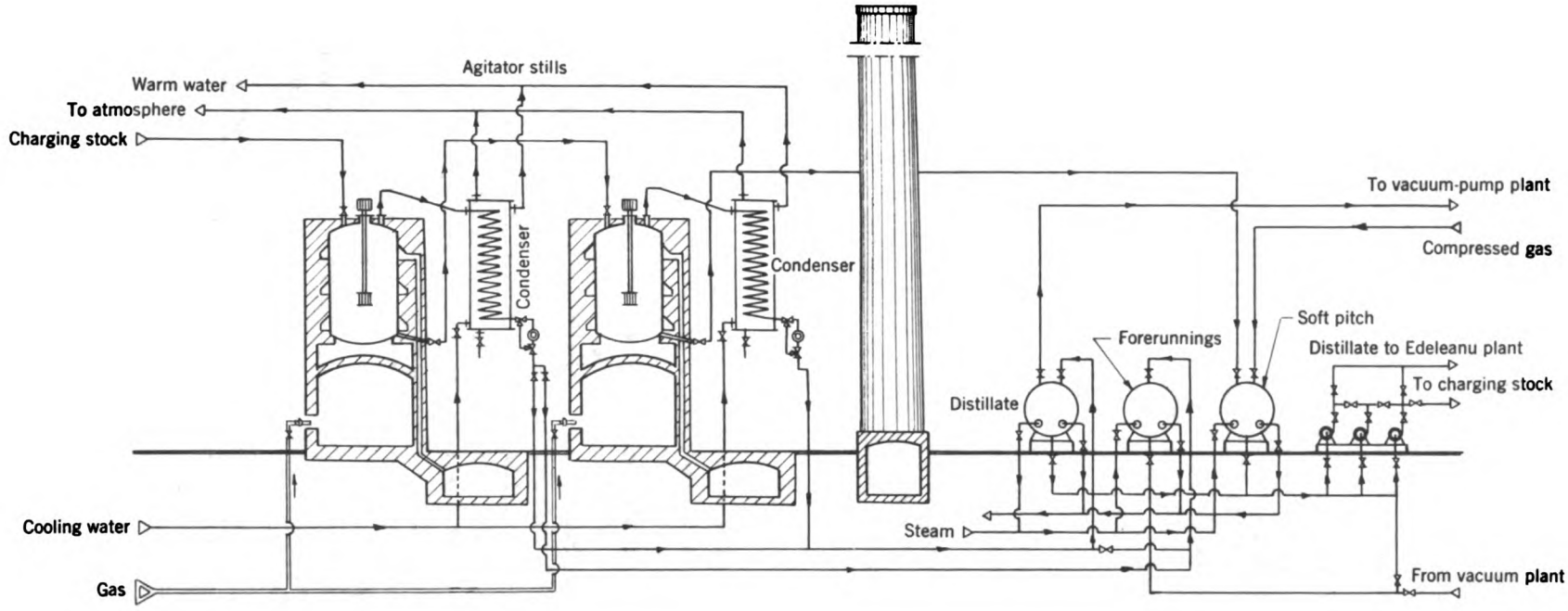

Figure 25. - Arrangement for redistillation of oil, A. G. Sächsische Werke, Espenhain.

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458) shows the arrangement for a plant having one still and necessary auxiliaries. Vapors from the stills are condensed with coil condensers, and the condensates flow by gravity to receivers. The distillation of the soft pitch is carried out principally by hot combustion gases bubbled through the pitch. Some heat is supplied by a gas burner beneath the still. The hot combustion gases are generated in the following manner: Gas, diluted with combustion gases from the combustion chamber of the still, is burned in a small vertical furnace. The oxygen-free combustion gases from this furnace, at a temperature of about 600° C. (1,112° F.), enter the still and are introduced below the surface of the pitch by means of a special distributing pipe. At the completion of distillation, the hard pitch is transferred by vacuum to a heated receiver, from which it can be transferred in liquid form to the pitch-coke ovens. The capacity of this part of the plant, using both stills, is about 40 metric tons of hard pitch per day.

Pitch carbonization: Frames 652-656.

The liquid, high-melting-point pitch is transferred to gas-heated feed tanks, from which it is pumped into separate regenerative coke ovens heated through the side walls by vertical flues (fig. 24, frame 656). The coke is pushed from each oven by a mechanical pusher and quenched with water. The coke is loaded into cars by means of a belt conveyor. The vapors from the ovens are condensed, and the condensate is added to crude brown-coal tar charged to the centrifugals and tar stills.

Oil redistillation: Frames 650-651.

The paraffin-containing distillates from the atmospheric and vacuum stills and from the hard-pitch still are combined and redistilled intermittently in gas-heated vertical stills equipped with agitators (fig. 25, frame 651). The latter are used to avoid overheating of the residues and to shorten the time of distillation. Several stills are employed for this purpose. The oil to be redistilled yields very little residue, so the distillations are conducted in such manner as to provide the minimum amount of residue needed in each still to avoid decomposition. A still containing partly distilled oil is charged with fresh oil. Atmospheric distillation is then used to remove light oil and part of the distillate. Vacuum is then used to remove additional distillate. The combined distillates from the atmospheric and vacuum distillations are transferred to the Edeleanu plant for wax extraction with sulfur dioxide. The residue from the vacuum distillation is returned to the stills, in which soft pitch is distilled to hard pitch by means of hot combustion gases.

Edeleanu Solvent Dewaxing. - The processes developed by Edeleanu Gesellschaft, Berlin, for the dewaxing of the paraffin-containing distillates from brown-coal tar are discussed at length in an article by Prof. Ernst Terres, Edeleanu-Gesellschaft, frames 739-753. They are also discussed and illustrated by means of a flow diagram in C. I. O. S. File XXVIII-23, Item 30 (PB 1,106), "A. G. Sächsische Werke-Espenhain, Kreis Borna, Germany", 1945, by G. S. Bays, J. P. Jones, and B. L. Mackusick, also available as a part of TOM Microfilm Reel 196. Briefly, the process, illustrated above in

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figure 18 was as follows: The redistilled oil from the tar plant containing paraffin wax was first extracted with SO_2. The extract was added to the paraffin-free oil from the tar plant, and the mixture was extracted with benzine to obtain Diesel oil and heating oil. The slurry resulting from the SO_2 extraction of the wax-containing distillate was filtered in a special band filter, in which the wax was washed with dichloroethane to remove contaminating oils and obtain raw hard wax, which, after acid and clay treating, yielded purified hard wax. The filtrate from the filtering operation mentioned above was cooled to -20° C. (-4° F.) and again filtered and washed in the band filter with dichloroethane. The raw soft wax recovered by this operation was clay-treated to produce finished soft wax, and the filtrate was added to the Diesel oil produced by the benzine extraction of the SO_2 extract and paraffin-free oil mixture. Figure 26 is an illustration of the band filter used to remove the hard wax. A description of this filter and its operation is given on pages 56 and 57. According to Prof. Terres, the over-all yield in weight percentages of products from the crude, low-temperature, brown-coal tar treated in the manner described above was as follows:

Product	Percent
Benzine and water	1.2
Hard paraffin	10.5
Soft paraffin	4.9
Diesel oil	33.3
Heating oil	34.7
Electrode coke	7.0
Gas and loss	8.4

The products made in the entire plant of A. G. "Sächsische Werke, Espenhain, in 1944, were reported in C. I. O. S. File XXVIII-23, Item 30 (PB 1,106), "A. G. "Sächsische Werke-Espenhain, Kreis Borna, Germany", 1945, by G. S. Bays, J. P. Jones, and B. L. Mackusick; also available as a part of TOM Microfilm Reel 196, to be as follows:

Product	Metric tons
Brown-coal briquets	2,696,000
Tar from low-temperature plant.	297,000
Coke from low-temperature plant	1,400,000
Fuel oil	42,778
Diesel oil	14,699
Hard wax	6,541
Soft wax	4,676
Electrode coke	7,080
25 percent crude phenols	32,000
Sulfur	22,000
Carbolic acid	9,600

Tar Distillation Process Developed by Byk-Guldenwerke, Chemische Fabrik A. G.

Byk-Guldenwerke Chemische Fabrik A. G., Berlin-Oranienburg, was said to have developed a method of working up low-temperature tars produced from

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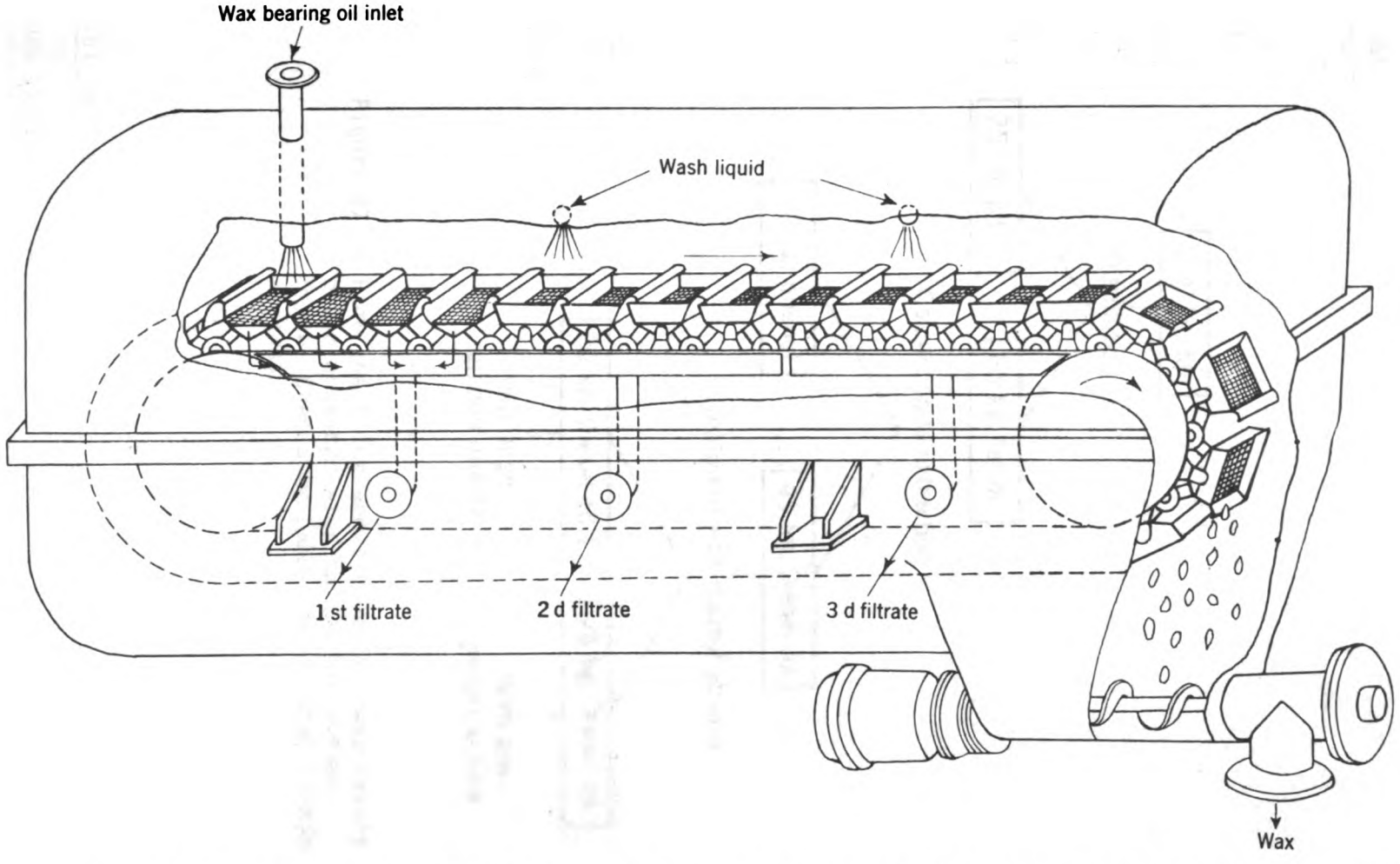

Figure 26. - Wolf continuous-band-cell filter for removing hard wax from solvent-treated, brown-coal-tar distillate at Espenhain, Edeleanu G. m. b. H., Altenburg.

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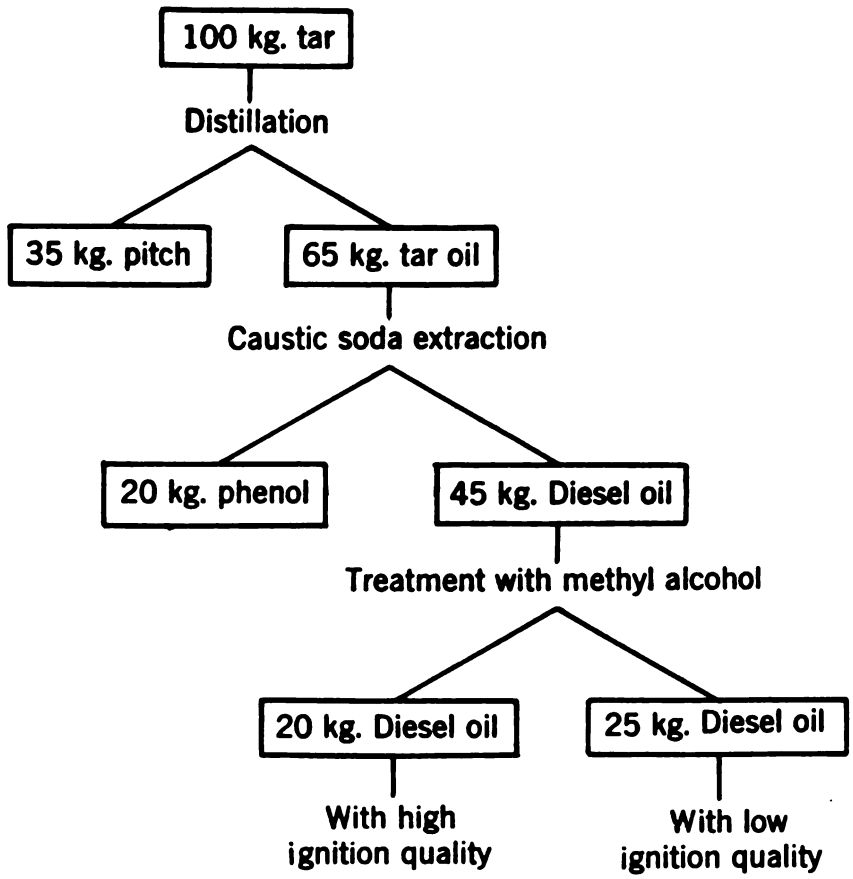

Figure 27. - Flow sheet for working up low-temperature, bituminous-coal-tar by the Byk-Gulden Werke, Chemische Fabrik A. G. distillation process.

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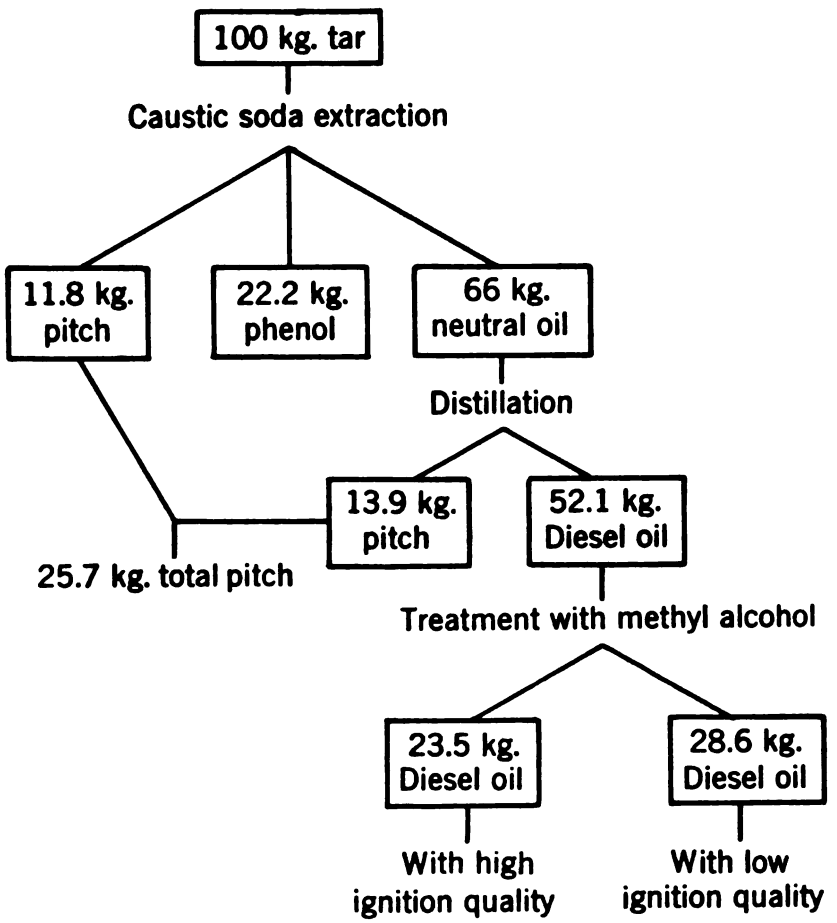

Figure 28. - Flow sheet for working up low temperature, bituminous-coal-tar by the Byk-Gulden Werke, Chemische Fabrik A. G., washing process.

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caking bituminous coal by the Brennstoff Technik process, frames 658-661; 484-485; 502-504. Two alternate methods of operation are illustrated by the flow diagrams in figures 27 and 28 (frames 503 and 504). In one process (fig. 27) the tar is distilled to obtain pitch and oil. The oil is extracted with caustic soda to remove phenols and to obtain Diesel oil, which, by means of methyl alcohol, is separated into two grades of Diesel oil of different ignition quality. In the other process (fig. 28) the tar is first mixed with light oil from the low-temperature carbonization process. The mixture of light oil and tar is then washed with caustic soda to extract phenols and obtain pitch and neutral oil. The neutral oil is then distilled, yielding pitch, which is combined with that from the caustic-soda extraction, and Diesel oil, which is separated by methyl alcohol as before into two grades of Diesel oil.

Experiments were conducted with these processes in a pilot plant at Oranienburg, near Berlin, from 1942 to 1944, and plans were formulated for the construction of large plants in the Saar and in Upper Silesia. However, construction of the large plants had not started, and the experimental plant had been destroyed by air raids when the war ended.

Pintsch Gas Plants:

The production of gas from brown-coal tar was carried out in 26 plants built by Julius Pintsch Kommanditgesellschaft, Berlin. The gas produced in these plants was used by the German railroads for the illumination of passenger cars and for the heating of car wheels in repair shops.

A typical plant of the Pintsch type was visited at Rummelsburg near Berlin. It had three gas-making machines. Each consisted of a preheater, two checker-filled generators, and tar-collecting equipment. The brown-coal tar passed first through the heat exchanger, where it was preheated by gases leaving the generators. A portion of the preheated tar then was burned in one of the generators to heat that generator to gasifying temperature while the remainder of the tar was being gasified in the other generator. At regular intervals the operations of the two generators were reversed. The generators operated at approximately 800°-850° C. (1,472°-1,562° F.) and 500 mm. (19.7 inches) water pressure.

The gasification of 1 metric ton of tar required burning 200 kilograms (441 pounds) of tar to heat the generators. It produced 250 to 500 cubic meters (8,830 to 17,660 cubic feet) of gas having a heating value of 9,000 kg.-calories per cubic meter (1,010 B.t.u. per cubic foot), and 30 kilograms (66 pounds) of light hydrocarbon oil. The latter was shipped to Rütgerswerke A.G. for refining. The gas was compressed to 15 atmospheres (220 pounds per square inch) for distribution through pipe lines to various points of use or redistribution.

The annual production of Pintsch gas from brown-coal tar in Germany was about seven to ten million cubic meters (247 to 353 million cubic feet). An additional one to two million cubic meters (35.3 to 70.6 million cubic feet) was produced in the same manner from petroleum oils.

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Yields of Products by Different Operating Procedures

The different yields of products that were obtainable from a single tar by working up the tar in different ways are shown in table 3, which was prepared from data published by Lurgi (frame 539). A brown-coal tar from the Lurgi-Spülgas process was subjected to (1) distillation with paraffin recovery, (2) pressure distillation, (3) cracking, and (4) hydrogenation.

TABLE 3. - Products from Lurgi-Spülgas brown-coal tar when processed by different methods

Products	Distillation and paraffin recovery, percent		Pressure distillation, percent	Cracking, percent	Hydrogenation, percent	
	A	B			A	B
Motor spirit	6	6	10	33-35	80	20
Diesel oil	21	35	45-50	20-22	-	39
Fuel oil	43	29	10	-	-	-
Lubricating oil..	-	-	-	-	-	17.5
Paraffin	9	9	-	-	-	13.5
Pitch	2	2	15-20	30	-	-
Electrode carbon.	9	9	-	-	-	-
Gas loss	10	10	15	15	20	10

Characteristics of German Low-Temperature Tars

Little information was obtained during the course of this investigation concerning the characteristics of German low-temperature tars that was not already contained in publications issued before World War II.

Apparently little investigational work dealing with the chemical constituents of low-temperature tar was carried out after 1939. In that year, Dr. O. Kruber of Gesellschaft für Teerverwertung m.b.H., published an article entitled "Beiträge zur Kenntnis des Steinkohlenschwelteeröls" in Oel und Kohle vereinigt mit Petroleum, volume 45, page 770 (frames 663-667).

For purposes of comparison, some analytical data for several German low-temperature tars are presented in table 4. The data were taken from documents collected in the course of the investigation.

Dephenolization of Low-Temperature Carbonization Waste Waters

An important aspect of low-temperature carbonization in Germany was the considerable effort given to removing phenols from waste waters. Profitable recovery of these phenols was generally secondary to keeping them from polluting rivers and streams.

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TABLE 4. - Characteristics of some low-temperature tars

Source of tar	Specific gravity at °C.	Solidifying point, °C.	Degrees Engler viscosity, at °C.	Flash point, °C.	Sulfur, percent	Ash, percent	Engler distillation, percent by volume					Source of information, frame No.
							To 200°C.	200°-230°C.	230°-270°C.	270°-300°C.	300°-360°C.	
Borsig-Geissen (brown coal)	0.905 to 0.960 at 60°C.	37	1.3 to 1.6 at 100°C.				2	6	12	10	25 (355°)	384 385
Lurgi-Spülgas (brown coal)	0.9175 at 60°C.	37		125	1.8		1	1.5	14.5	12.5	12.5 (320°)	385
Lurgi-Spülgas (bituminous, high-volatile coal)	1.055 at 20°C.	20.5	7.7 at 50°C.			0.15	2	11	15	10	24	388
Krupp-Lurgi (bituminous coal - Ruhr)	1.05 to 1.06	-15 to -20	2 at 50°C.	60 to 70	0.7	Less than 0.15	6.7	6.4	10.9	8.7	27	389
Brennstoff-Technik 520°-560° C. (Bituminous coal)	1.034 at 15°C.					0.05	4.7	13.1	15.9	8.9	23.6	490

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The dephenolization of waste waters from low-temperature carbonization plants is a somewhat more difficult problem than that encountered at high-temperature plants. In addition to the phenol, cresols, and xylenols occurring in high-temperature carbonization waste waters, the low-temperature plant waters also have higher-boiling alkylated phenols, dihydroxy benzenes, and fatty and humic acids. Of the several dephenolizing processes used in Germany, not one seems to have sufficient flexibility to cover high- and low-temperature carbonization and hydrogenation-plant waste waters. The various processes employed will be reviewed briefly from the standpoint of their application to low-temperature carbonization waters.

Dephenolization by Destruction of Phenols: Frame 994.

(a) Evaporation and then burning of the residue. Corrosion was a particularly bad problem in this process. In one installation, all waste water was evaporated directly into the air by gas-heated furnaces. Thereby, most of the phenols were burned before leaving the 250-foot high stack of the furnace.

(b) Quenching of coke with the waste waters. This method merely resulted in polluting the atmosphere. Coke of inferior quality was obtained.

(c) The waste water was used at coal-flotation plants in Lower Silesia, where its frothing characteristics appeared useful.

(d) Aeration and bacteriological oxidation, with phosphates to promote bacterial growth, was practiced by Emschergenossenschaft and others. This process was generally not a primary step but was used to clean up waters already partially dephenolized.

Dephenolization by the Benzene Process: Frames 928-983; 995; 1016-1018.

The original process consisted of washing the waste waters with benzene and recovering the benzene for recycling by distillation. A very large volume of benzene was required for substantial phenol removal. Recovery of this volume of benzene by distillation was costly. Emschergenossenschaft, which originally used the process, changed to the Pott-Hilgenstock variation (fig. 29, frame 995), wherein the benzene washings were continuously extracted with caustic soda solution. Phenols were separated as phenolates, and the benzene was recycled. This process was used in the Ruhr coking plants, where phenolates were centrally treated for phenol recovery. Its application to low-temperature carbonization-plant waters was limited. Benzene was not a natural byproduct of such plants and therefore had to be shipped in. Any losses due to the high solubility of benzene in the waste water had to be strictly watched. This necessitated high-steam stripping costs for benzene recovery from the extracted water. A further variation of this extraction process involved the use of dephenolized middle oil from low-temperature carbonization plants in place of benzene. The water solubility of this oil was less than that of benzene. However, frequent difficulties were encountered owing to stubborn emulsions. Again, as with the Pott-Hilgenstock benzene process, phenolates were formed from low-temperature carbonization

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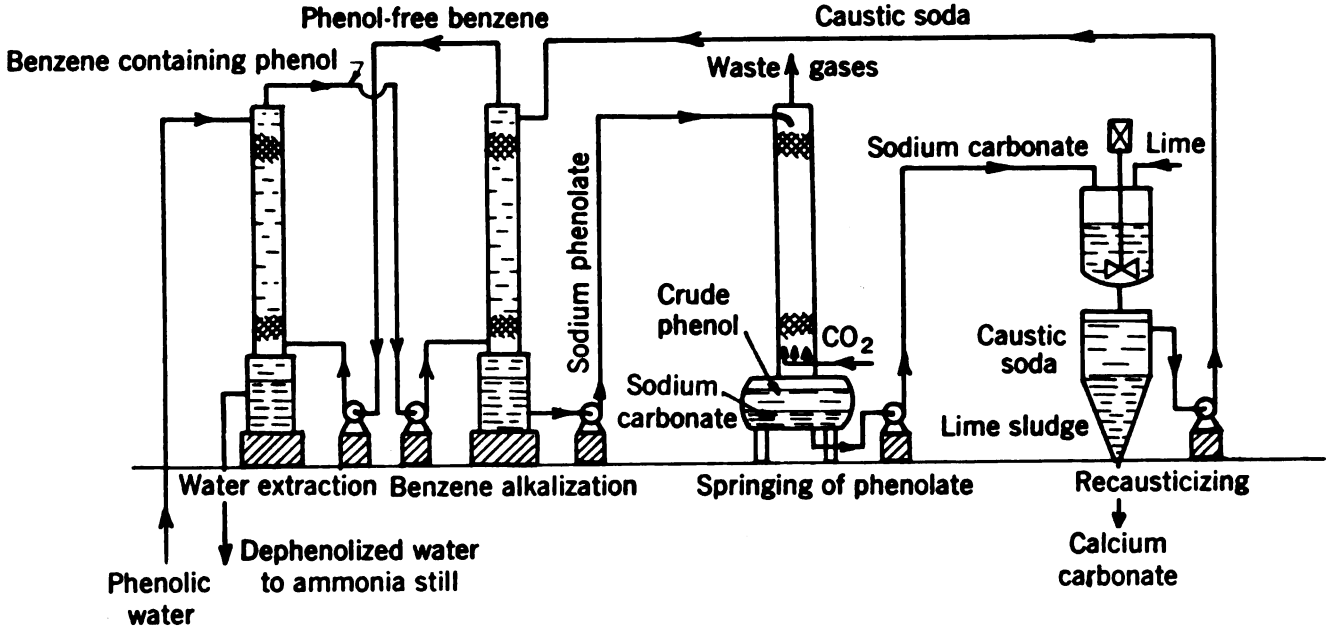

Figure 29. - Flow sheet of the Pott-Hilgenstock process for obtaining phenols from waste waters of coke-oven plants.

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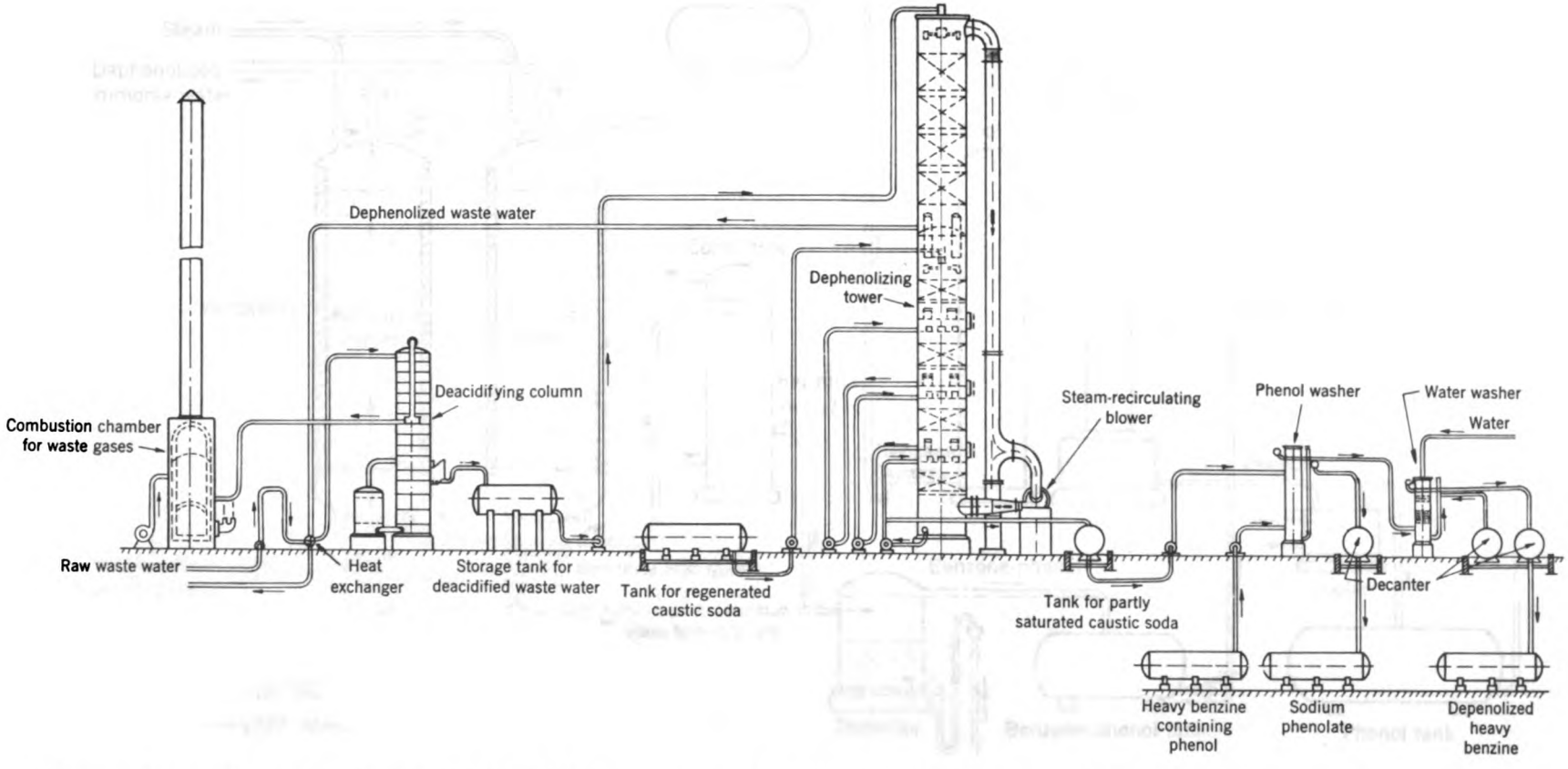

Figure 30. - Koppers steam-recirculating process for dephenolizing brown-coal-carbonization, waste water, A. G. Sächsische Werke, Böhlen.

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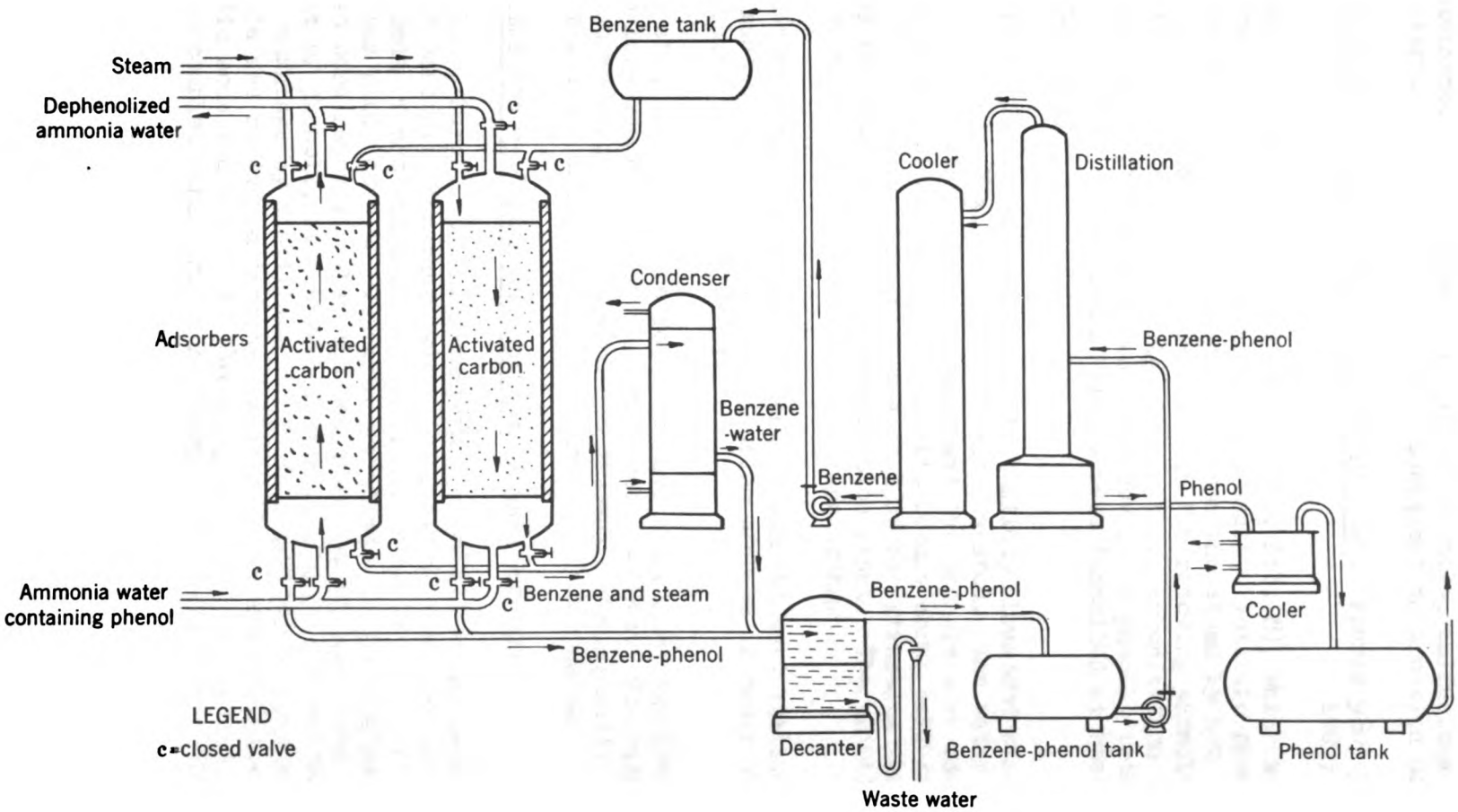

Figure 31. - Activated-carbon process for dephenolizing waste water from coke-oven plants, Carbo-Norit-Union and Lurgi.

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liquors which could not be completely sprung with CO_2. To recover more dihydroxy benzenes and the higher alkylated phenols, sulfuric acid treatment was necessary. This resulted in the subsequent destruction of soda.

Dephenolization by the Steam-Stripping Process: Frames 980; 981; 996; 1007-1009; 1012.

In this process, figure 30 (frame 1012, Abb. 8), which was developed by Koppers in 1925-28, no solvent is necessary. Volatile phenols are stripped with steam and the phenols are removed from the steam by hot caustic soda. This process was used before the last war for removing phenols from ammoniacal liquors at some of the German high-temperature carbonization plants. During the war it was adapted as well to the recovery of phenols from low-temperature carbonization waste water. Plants were installed at Most (Brux), Böhlen, and Espenhain.

At these plants the water was first mildly blown with steam to remove CO_2, H_2S, and some NH_3. Some phenol also escaped. The water was then countercurrently stripped with steam, and the phenols were caught in hot caustic-soda solution. After the caustic became about half-saturated with phenols, its residual alkalinity was used up by extracting phenol-bearing oils from the low-temperature carbonization plant. The phenolate, as in the benzol method, was processed by CO_2 springing.

Low-temperature plant waters contain some steam-volatile fatty acids. Ammonia must be maintained in the steaming process in order to decrease the volatilization of the fatty acids.

The steam stripping was found to have a distinct disadvantage when applied to low-temperature carbonization waste waters. The higher boiling phenols, pyrocatechol, etc., were not removed. Consequently, additional treatment was necessary to make the waste water innocuous.

Dephenolization by Carbon Adsorption: Frames 996-997.

In 1930, Lurgi, at a high-temperature carbonization plant in the Ruhr, clarified carbonization waste waters by passing them through coke and then through beds of activated carbon (fig. 31, frame 997, Abb. 4). The phenol removal was good, down to 0.05 gram per liter. Higher-boiling phenols and oils also were removed. When the activated carbon had taken up 6-10 percent by weight of phenols, it was washed with benzene. The benzene was removed from the washings by distillation. By this process a greater percentage of phenols was recovered than by the benzol or steam-stripping methods, and no caustic soda was required. Steaming of the carbon served to reactivate it and to complete the benzene recovery. The high-phenol recoveries would appear to make the process attractive. However, the plant was shut down when phenol prices dropped, and no further operations were started, possibly for the following reasons: (a) The process was not continuous; (b) reactivation of the carbon was difficult owing to the accumulation of high-boiling acidic and tarry constituents; and (c) plants had already been constructed for working up phenolates from the Pott-Hilgenstock steam-stripping and other processes.

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Dephenolization by the Tricresylphosphate Process: Frames 992; 997; 1006-1007.

I. G. Farbenindustrie A. G. developed a solvent-washing process which appeared to have promise for removing phenols from hydrogenation and low-temperature carbonization plant waters (fig. 32, frame 992). The solvent is tricresylphosphate, "Triphos." It has a very high boiling point and a high adsorption coefficient for phenols. Phenolic waters extracted with 15-20 percent tricresylphosphate will have a residual phenol content of about 0.1 gram per liter.

Phenols are recovered from the solvent extract by vacuum distillation. Tricresylphosphate remains as a distillation residue ready for recycling. Cleaning up of the tricresylphosphate with fuming sulfuric acid and lime is necessary when accumulations of tarry and high-boiling materials occur.

Several plants were erected, but many disadvantages became apparent, especially in dephenolizing low-temperature carbonization-waste waters. High-boiling phenols were very difficult to remove by distillation. These accumulated in the tricresylphosphate and decreased its dephenolization efficiency. Moreover, this accumulation of high boilers, tars, and phenols thickened the tricresylphosphate, with subsequent emulsification troubles. Frequent and costly sulfuric acid purifications of the solvent were necessary. The process is better suited for hydrogenation and high-temperature coking-plant waste waters.

Dephenolization by the Phenosolvan Process: Frames 998-1004.

As more and larger low-temperature coal-carbonization plants were built, the need for a good dephenolizing scheme became more urgent. A large number of solvents was studied.

A method developed by Metallgesellschaft A. G. for cleaning up hydrogenation waste liquors was found to be applicable to low-temperature carbonization waters, as, for example, from the Lurgi-Spülgas process.

The method makes use of a special solvent called "phenosolvan." This solvent, although frequently referred to as a mixture, is essentially isobutyl acetate. It is nontoxic, has a boiling point of 110°-130° C. (230°-266° F.), and a specific gravity of 0.88. Claims are made that no emulsification difficulties are encountered in the use of phenosolvan and that the solvent losses are low, i.e., 0.1 to 0.2 gram per liter.

The following extraction efficiencies are given for low-temperature carbonization waste waters. To reduce the phenol content from 7.0 grams per liter to 0.1 gram per liter requires, in volume percent, either 220 of benzene, 250 of dephenolized low-temperature carbonization oil, 17.5 of tricresylphosphate, or 10 of phenolsolvan.

In 1940, Lurgi and I. G. Farbenindustrie A. G. collaborated in the commercial exploitation of this process (fig. 33, frame 1010; and fig. 34, frame

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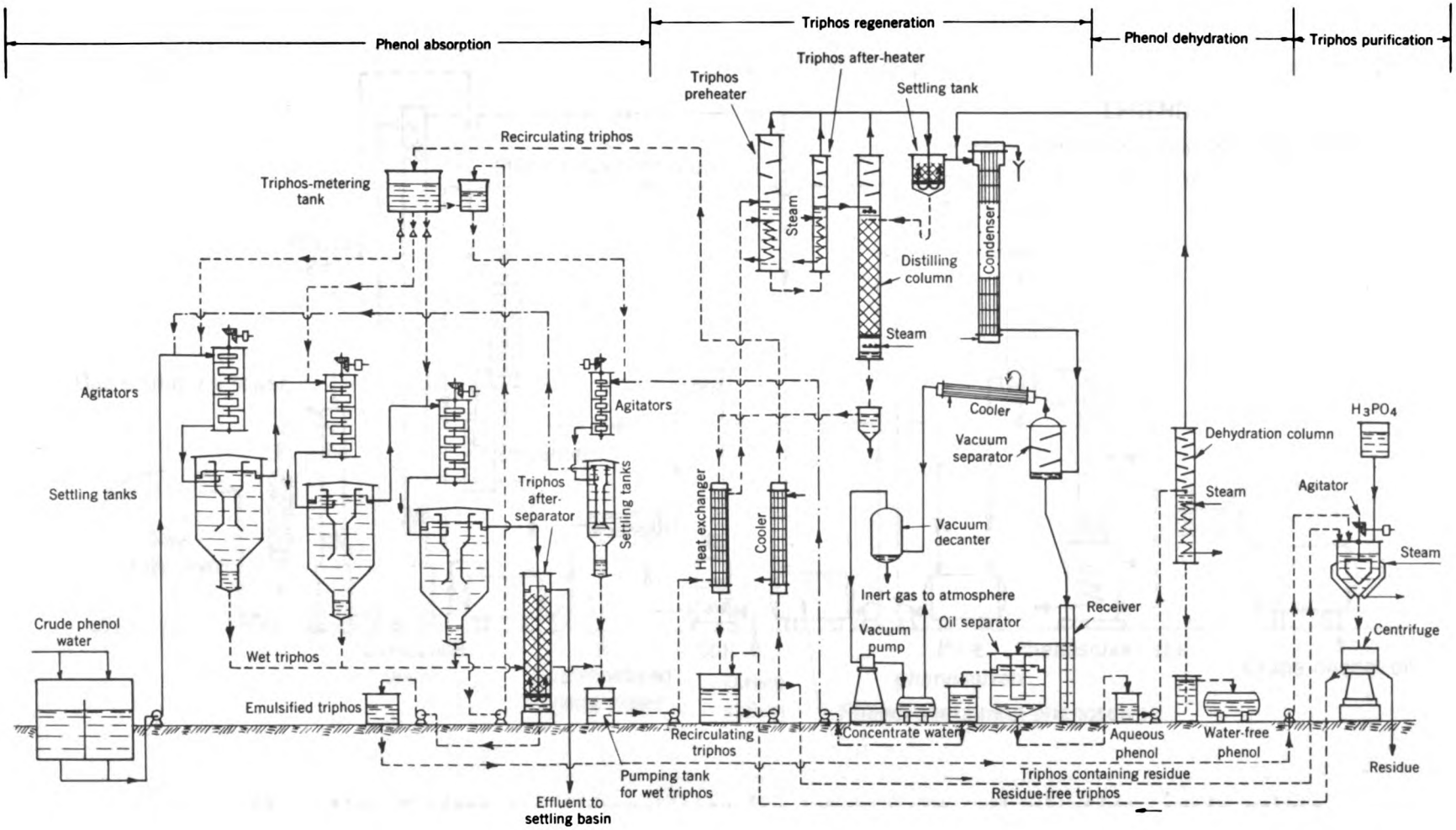

Figure 32. - Tricresylphosphate process for dephenolizing plant-waste waters ("Triphos" method), I. G. Farbenindustrie A. G., Merseburg-Leuna.

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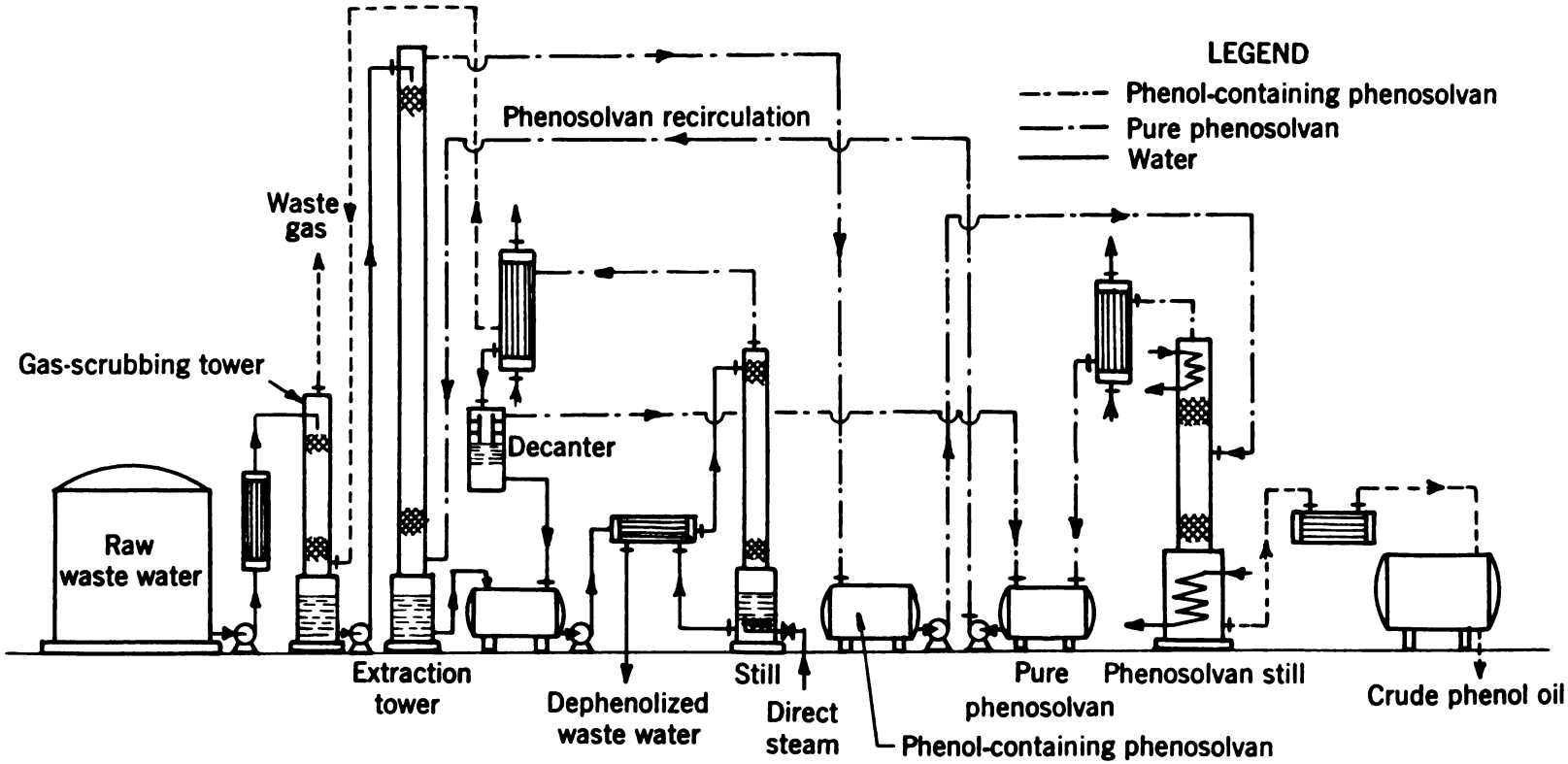

Figure 33. - Flow diagram for dephenolizing low-temperature carbonization, waste waters by the phenosolvan process, Lurgi.

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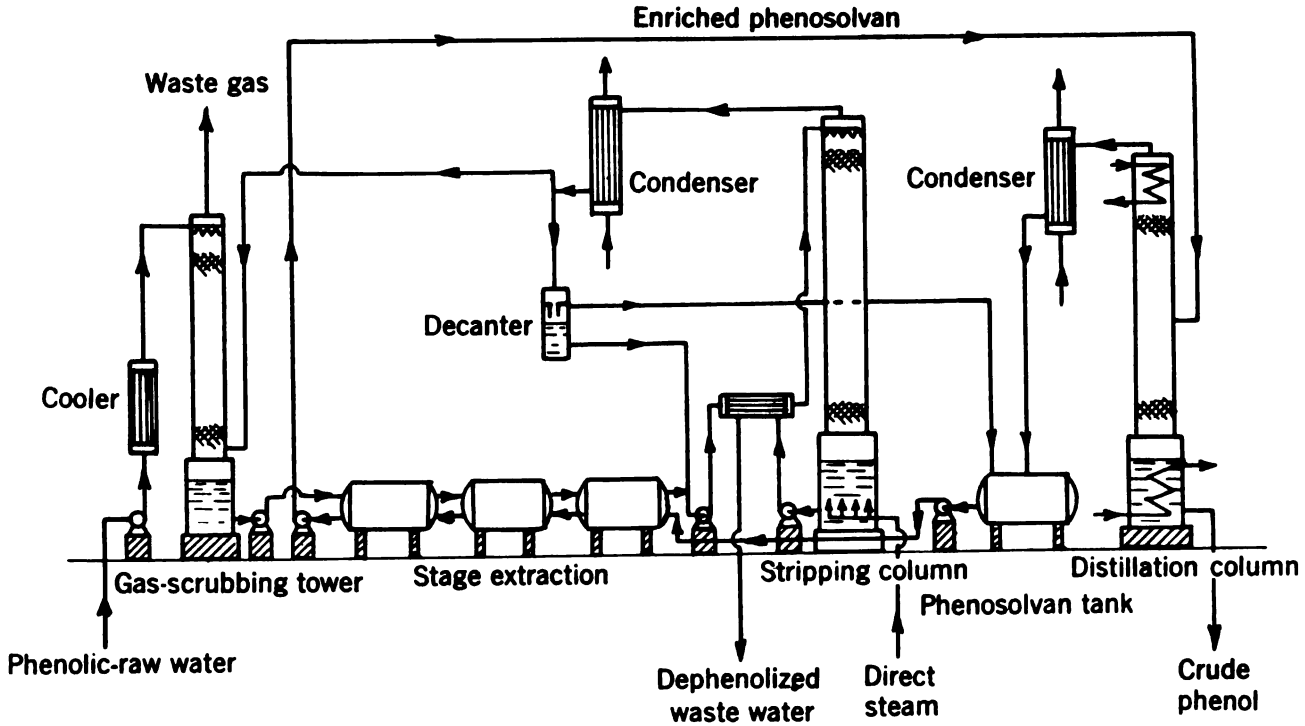

Figure 34. - Flow diagram of phenosolvan plant for phenol extraction from hydrogenation and low-temperature-carbonization, waste waters, I. G.-Lurgi process, Sudetenländische Treibstoffwerke, Most (Brüx).

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998, Abb. 6). The first large plant was put into operation on hydrogenation waste waters at Pölitz, near Stettin. The process was apparently considered successful, as soon thereafter a phenosolvan plant was set up at Blechhammer and another at Most (Brüx) for treating low-temperature carbonization waters and hydrogenation liquors. A fourth was being constructed in Saarbrücken at the end of the war. Two other phenosolvan plants were built by Lurgi in 1947 - one at the Brabag works at Böhlen and another at Leuna. Both were for the dephenolization of hydrogenation liquors.

The low-temperature waste waters first had the tar and oils removed mechanically. The water was then treated in a three-stage countercurrent extraction system with the equivalent of 10 percent phenosolvan. About 0.7 percent phenosolvan remained dissolved in the treated water. This was recovered by steam stripping and was reused. The phenosolvan extract was continuously distilled, whereby distilled phenosolvan was recovered for recycling. Mixed phenols were recovered as a distillation residue to be processed later for phenol, cresols, xylenols, and pyrocatechol, as, for example, at the Leuna works.

At Blechhammer there were serious restrictions on effluent disposal, and, although the phenosolvan process reduced the tar-acid content of the spent liquor to less than 200 milligrams per liter, further treatment was necessary. The liquor was diluted, heated to 25° C. (77° F.), treated with bacteria, and pumped over waste ashes from the power station. The final tar-acid content was less than 0.5 milligram per liter.

Refining and Use of Tar Acids from German Low-Temperature Carbonization and Hydrogenation Plants

The largest plant in Germany for working up the tar acids recovered from brown-coal carbonization and hydrogenation plants was at Leuna near Merseburg. It was owned and operated by I. G. Farbenindustrie.

The monthly tonnages of crude tar acids received at Leuna from various sources and the monthly quantities of refined tar acids produced at that plant during the period January to May 1944 are given in table 5.

The extremely diversified and extended operations of the I. G. Farbenindustrie Leuna plant are herein considered only from the standpoint of the processing and partial utilization of tar acids. Crude and semirefined phenols were received at the Leuna South Phenol Works from the Leuna liquid-phase hydrogenation-plant middle oil, from low-temperature carbonization and hydrogenation plant waste waters, and from other sources. Tar acids from the waste-water sources had been extracted by the phenosolvan, tricresylphosphate, and Koppers steam-stripping dephenolization processes at Leuna, Most (Brüx), Pölitz, Blechhammer, A. S. W., Brabag, and elsewhere. These tar acids were all brought into the Leuna South Phenol Plant, where, after some preliminary treatment, they were combined and processed together.

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TABLE 5. - Tar acid received and produced per month at Leuna plant of I. G. Farbenindustrie, January to May, 1944 1/

	Metric tons, per month
Sources of crude-tar acid:	
Oils from brown-coal hydrogenation at Leuna	870
Phenosolvan extracts from hydrogenation and low-temperature carbonization waste water:	
Pölitz	322
Blechhammer	163
Most (Brüx)	1,152
	2,507
Production of refined-tar acids:	
38° C. (100.4° F.) m.p. phenol	15
40.2° C. (104.4° F.) m.p. phenol	610
Ortho fraction	100
Ortho cresol (50 percent)	90
Cresol DAB4 (at least 38 percent m-cresol)	360
Cresol DAB6 (at least 50 percent m-cresol)	40
Xylenol	230
Xylenol N	70
Crude pyrocatechol I	25
Crude pyrocatechol II	130
Crude pyrocatechol III	2
Crude pyrocatechol IV	70
Residues	710
	2,452

1/ Crude-tar acids from A. S. W., Brabag, and elsewhere were also processed at times in the Leuna plant.

Processes and products that will be covered briefly in this section are (1) tar-acid extraction and processing at the Leuna coal-hydrogenation plant, (2) pretreatment of the separately derived crude-tar acids, (3) fractional distillation of refined tar acids, (4) sulfuric-acid refining of crude cresol, (5) production of pyrocatechol, (6) hydrogenation of phenol to cyclohexanol, (7) production of cyclohexanone, (8) manufacture of cyclohexanone oxime and caprolactam, (9) manufacture of adipic acid, and (10) manufacture of Igamids.

Numerous references are given in the text to plant and equipment layouts and descriptive literature. These references are indicated by frame numbers, which refer to FIAT Microfilm Reel C-98 or TOM Microfilm Reel 152 (PB L 13,093).

Tar-Acid Extraction and Processing at the Leuna Coal-Hydrogenation Plant: Frames 678-684.

The tar-acid processes employed in this plant were (a) extraction of tar-acid oil and purification of the resulting phenolate, (b) transfer to the CO_2 and caustic plant for springing of tar acids, and (c) final springing

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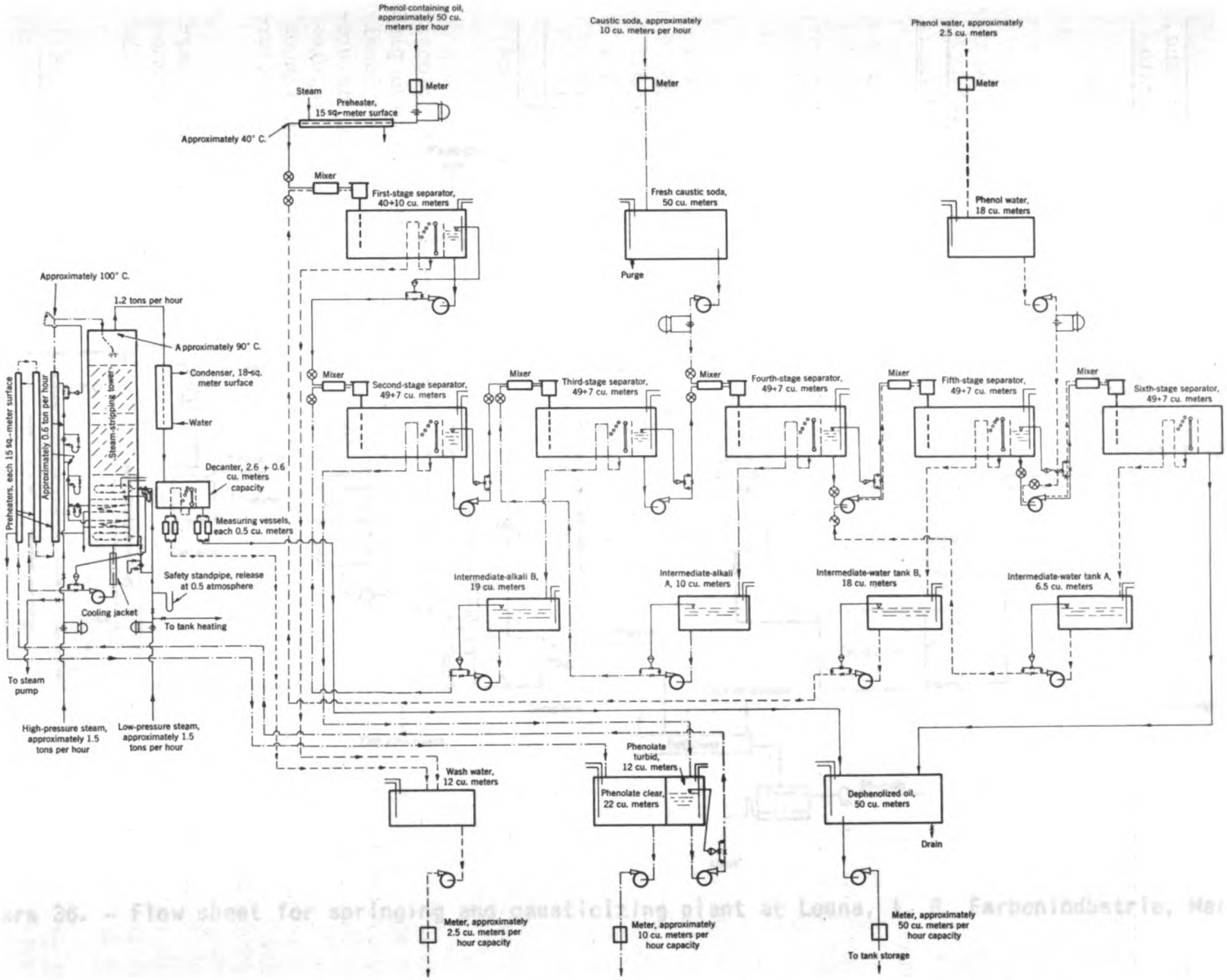
Phenol-containing oil, approximately 50 cu. meters per hour
Meter
Steam
Preheater, 15 sq-meter surface
Approximately 40° C.
Mixer
First-stage separator, 40+10 cu. meters
Caustic soda, approximately 10 cu. meters per hour
Meter
Fresh caustic soda, 50 cu. meters
Purge
Phenol water, approximately 2.5 cu. meters
Meter
Phenol water, 18 cu. meters
Approximately 100° C.
1.2 tons per hour
Approximately 90° C.
Condenser, 18-sq. meter surface
Water
Steam-stripping tower
Approximately 0.6 ton per hour
Preheaters, each 15 sq.-meter surface
Decanter, 2.6 + 0.6 cu. meters capacity
Measuring vessels, each 0.5 cu. meters
Safety standpipe, release at 0.5 atmosphere
Cooling jacket
To tank heating
To steam pump
High-pressure steam, approximately 1.5 tons per hour
Low-pressure steam, approximately 1.5 tons per hour
Mixer
Second-stage separator, 49+7 cu. meters
Mixer
Third-stage separator, 49+7 cu. meters
Mixer
Fourth-stage separator, 49+7 cu. meters
Mixer
Fifth-stage separator, 49+7 cu. meters
Mixer
Sixth-stage separator, 49+7 cu. meters
Intermediate-alkali B, 19 cu. meters
Intermediate-alkali A, 10 cu. meters
Intermediate-water tank B, 18 cu. meters
Intermediate-water tank A, 6.5 cu. meters
Wash water, 12 cu. meters
Phenolate turbid, 12 cu. meters
Phenolate clear, 22 cu. meters
Dephenolized oil, 50 cu. meters
Drain
Meter, approximately 2.5 cu. meters per hour capacity
Meter, approximately 10 cu. meters per hour capacity
Meter, approximately 50 cu. meters per hour capacity
To tank storage

Figure 35. - Arrangement of a continuous tar-acid extraction plant at Leuna, I. G. Farbenindustrie, Merseburg.

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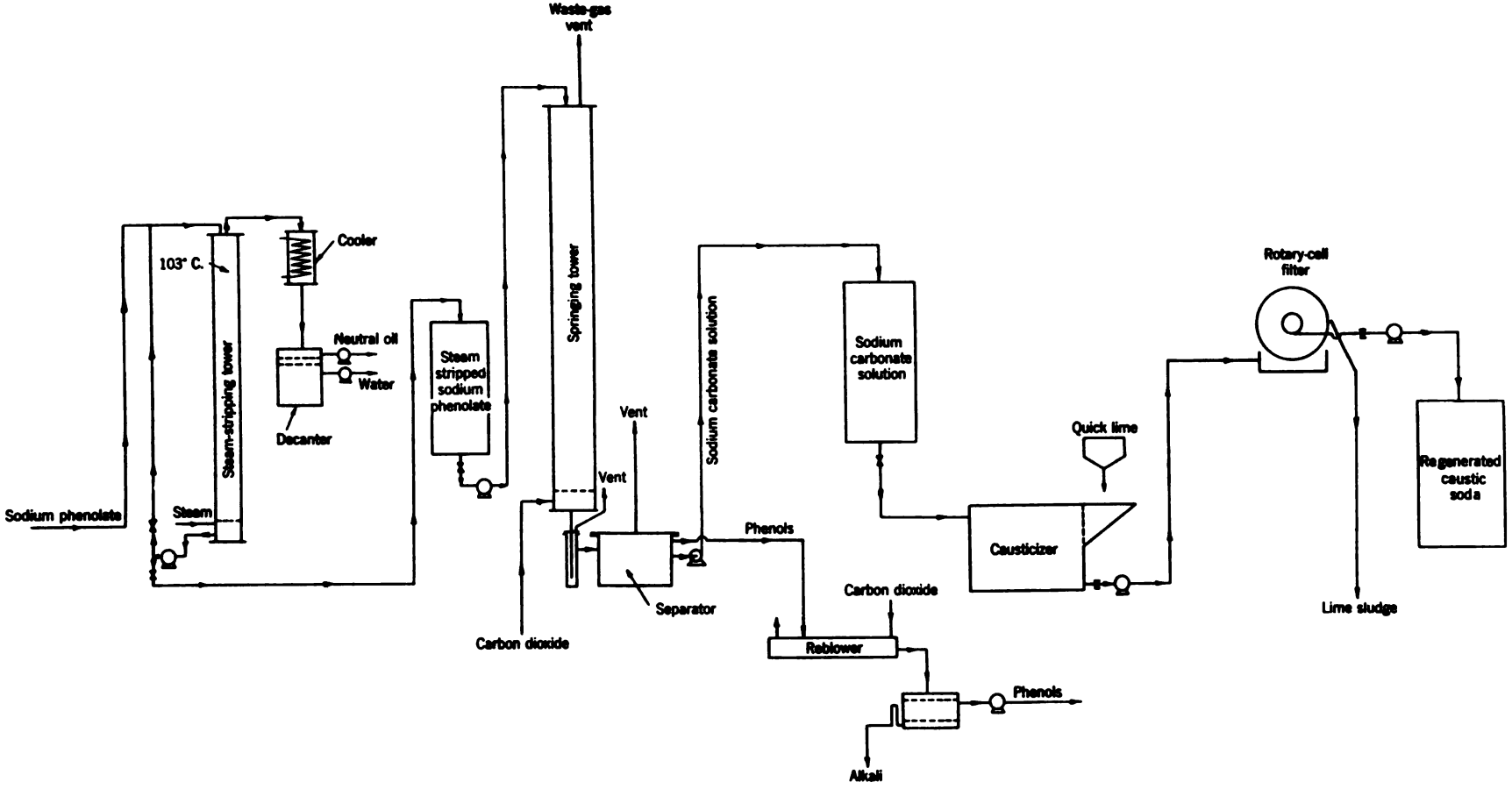

Figure 36. - Flow sheet for springing and causticizing plant at Leuna, I. G. Farbenindustrie, Merseburg.

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and primary distillation of the tar acids after their return from the CO_2 plant. The distilled tar acids were sent to the Leuna South Phenol Plant for further refining.

(a) Extraction and Phenolate Purification:

Distillate middle oil from the coal-hydrogenation plant, containing about 15 percent tar acids, was extracted in a six-stage, continuous, tar-acid-extraction system. The arrangement of the equipment is shown in figure 35 (frame 682). Mixing was done in chambers equipped with orifice and baffle plates. After leaving the chambers, the mixtures were continuously separated in 14,800-gallon (56-cubic meter) tanks partitioned into 12,950-gallon (49-cubic meter) and 1,850-gallon (7-cubic meter) sections, except for the first-stage 13,200-gallon (50-cubic meter) tank, which was partitioned into 10,560-gallon (40-cubic meter) and 2,640-gallon (10-cubic meter) sections. Oil overflowed from the larger section into the smaller one, from which it was taken to the next stage. The washings left the bottom of the larger section. Each stage had its mixing chamber and partitioned decanting tank.

The oil was treated at 40° C. (104° F.) in the first-stage washer with weakly alkaline phenol-wash water from the firth-stage washer. Next, the oil entered the second-stage washer with phenolate from the third stage. The oil was further extracted with phenolate in the third-stage washer and with 10 percent caustic soda in the fourth stage. A final clean-up of the oil was made in the fifth and sixth stages with phenol waste water.

The saturated phenolate from the second-stage washer was preheated to 100° C. (212° F.) and then steam-stripped of its oil in a tower. This tower was 43 feet high, had a diameter of 6.5 feet, and was filled with Raschig rings. Crude phenolate entered at the top of the tower and steam at the bottom. The oil-free phenolate leaving the bottom of the stripping tower contained 17 to 20 percent of tar acids. The oil and steam coming from the top of the column were condensed, the decanted oil going to dephenolized-oil storage and the water to phenol-waste water.

(b) Springing and Causticizing:

Purified phenolate was pumped to the carbonation and causticizing plant, where tar acids were liberated by CO_2 and the soda was recausticized. This plant was not a part of the hydrogenation works. A flow sheet of the plant is shown in figure 36 (frame 684).

(c) Final Springing and Primary Distillation of Tar Acids:

Crude tar acids from the preceding carbonation were returned to the hydrogenation-plant phenol works. These acids contained 11 to 12 grams of soda per liter. The high-alkali content was objectionable from the distillation standpoint, as it caused plugging of the preheaters and polymerization of the distillation residue. Consequently, additional springing and soda removal were necessary.

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The crude-tar acids were received at about 50° C. (122° F.) and were stored in a 9,000 gallon tank. Some carbonate settled out and was occasionally drawn off. Processing was continuous from this stage on through the distillation. Figure 37 (frame 683) shows a detailed flow diagram of the plant.

The tar acids were pumped into the top of a gassing tower consisting of six sections filled with Raschig rings. Before going into the tower, the tar acids were mixed with an equal quantity of 15 percent aqueous sodium sulfate solution. This admixture was claimed to be necessary to prevent crusts of solid sodium bicarbonate from plugging the gassing tower. The mixture, preheated to 75° C. (167° F.), entered the top of the tower and was contacted by a countercurrent stream of CO_2 entering at the bottom. The gassed mixture was pumped from a constant level in the bottom section of the tower. It went to a partitioned separation tank of 6,600 gallons capacity, from which the purified tar acids overflowed into a 13,200-gallon receiver. Settled bicarbonate-sodium sulfate solution from the partitioned separator was converted to sodium sulfate with 68 percent sulfuric acid. Acidification of the bicarbonate was controlled so as not to liberate the fatty acids from their sodium salts. Corrosion by fatty acids was thus avoided.

The cleaned-up tar acids, containing about 15 percent water, were preheated to 120°-125° C. (248°-257° F.) and drawn into a suction tank, where water and some forerunnings were flashed off, leaving tar acids with only about 3 percent water. The partly dried acids then entered the distillation system. In this there were three identical columns each consisting of four sections packed with Raschig rings, equipped with high-pressure steam coils at the bottom, and operating under a vacuum of 40-60 mm. (1.6-2.4 inches) Hg absolute. Tar acids from the suction tank were pumped through a preheater heated to about 120° C. (248° F.) and entered the top of the bottom section of the first column. Water and some phenols were flashed off the top of the column, and the residue at the bottom at 115°-125° C. (239°-257° F.) was pumped through a preheater heated to about 160° C. (320° F.) and into the second column. Here, a crude, high-phenol fraction was obtained overhead, and the residue, at about 160° C. (320° F.), was pumped into the third column, where a second tar-acid distillate was produced. The residue in the third column was held at about 160°-170°C. (320°-338° F.). It was used in the hydrogenation plant or shipped out of the plant. Distillates from this plant were further processed and blended in the Leuna South Phenol Plant.

The average throughput of the plant was 2.5 to 2.7 metric tons per hour of crude wet-tar acids.

Average composition of tar acids in the distillation system during October, November, and December 1943:

	Charge, percent			No. 2 column distillate, percent			No. 3 column distillate, percent		
	Oct.	Nov.	Dec.	Oct.	Nov.	Dec.	Oct.	Nov.	Dec.
Phenols	36.5	36.8	26.2	48.3	46.2	35.0	23.2	19.1	11.1
Cresols	36.8	39.2	38.3	38.5	42.0	44.0	48.5	49.0	39.2
Xylenols	6.7	9.8	15.8	4.0	4.2	12.5	15.6	23.5	26.0
Boiling higher than xylenols	20.0	14.2	19.7	9.2	7.6	8.5	12.7	8.4	23.7

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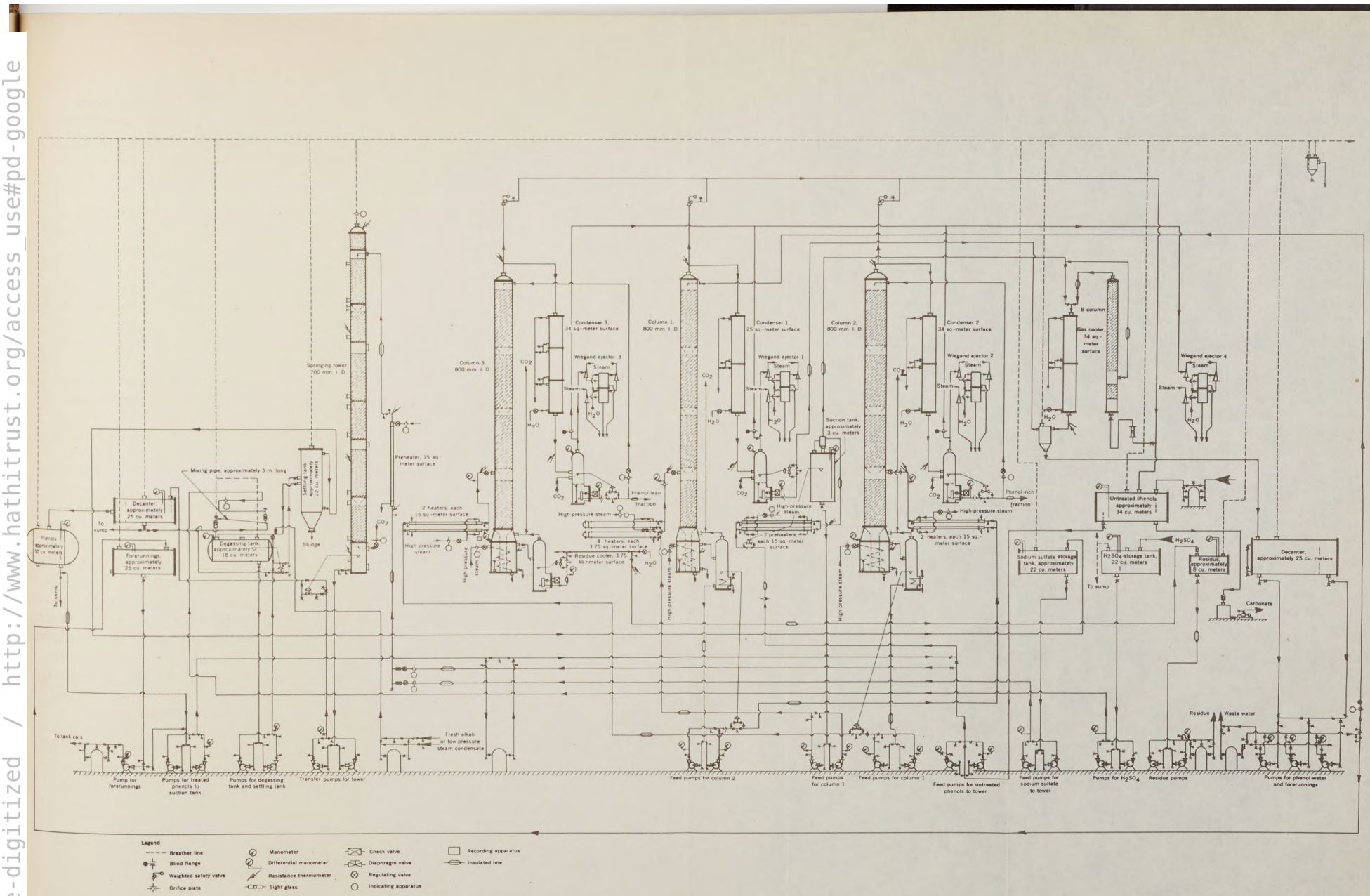
Springing tower 700 mm. I. D.
Column 3, 800 mm. I. D.
Column 1, 800 mm. I. D.
Column 2, 800 mm. I. D.
B column
Condenser 3, 34 sq.-meter surface
Condenser 1, 25 sq.-meter surface
Condenser 2, 34 sq.-meter surface
Gas cooler, 34 sq.-meter surface
Wiegand ejector 3
Wiegand ejector 1
Wiegand ejector 2
Wiegand ejector 4
Steam
Suction tank, approximately 3 cu. meters
Preheater, 15 sq.-meter surface
Mixing pipe, approximately 5 m. long
Settling tank, approximately 22 cu. meters
Sludge
1 Decanter, approximately 25 cu. meters
Phenols approximately 50 cu. meters
Degassing tank, approximately 18 cu. meters
Forerunnings, approximately 25 cu. meters
To sump
2 heaters, each 15 sq.-meter surface
High pressure steam
4 heaters, each 3.75 sq.-meter surface
Residue cooler, 3.75 sq.-meter surface
Phenol lean fraction
Phenol rich fraction
2 preheaters, each 15 sq.-meter surface
2 heaters, each 15 sq.-meter surface
Sodium sulfate storage tank, approximately 22 cu. meters
Untreated phenols approximately 34 cu. meters
H_2SO_4 storage tank, 22 cu. meters
Residue, approximately 8 cu. meters
Decanter, approximately 25 cu. meters
Carbonate
Residue
Waste water
To tank cars
Fresh alkali or low pressure steam condensate
Pump for forerunnings
Pumps for treated phenols to suction tank
Pumps for degassing tank and settling tank
Transfer pumps for tower
Feed pumps for column 2
Feed pumps for column 1
Feed pumps for column 1
Feed pumps for untreated phenols to tower
Feed pumps for sodium sulfate to tower
Pumps for H_2SO_4
Residue pumps
Pumps for phenol-water and forerunnings
Legend
Breather line
Blind flange
Weighted safety valve
Orifice plate
Manometer
Differential manometer
Resistance thermometer
Sight glass
Check valve
Diaphragm valve
Regulating valve
Indicating apparatus
Recording apparatus
Insulated line

Figure 37. - Flow diagram for final springing and primary distillation of tar acids at Leuna, I. G. Farbenindustrie, Merseburg.

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Pretreatment of Tar Acids

(a) Phenosolvan extracts: Frames 685; 690.

The phenosolvan extracts from Most (Brüx), Pölitz, and Blechhamer had the following approximate compositions:

	Percent		
	Most (Brüx)	Pölitz	Blechhammer
H_2O	0.5	0.5	0.5
Phenol	18	40	20
Cresols	13	20	20
Xylenols	5	5	5
Pyrocatechol and homologues	40	-	-
Higher boilers and residue	23.5	34.5	54.5

These extracts, as received at the tar-acid plant, could not be directly fractionated because of the very high percentage of high-boiling compounds. The heavy build-up of residue in the stills slowed down and at times even stopped the distillation, although only a small part of the available light phenols had been recovered. A preliminary clean-up distillation at only 1 to 1 reflux ratio, or less, was necessary in order to separate the bulk of the phenol, cresols, and xylenols. These were recovered as a refined tar-acid mixture and were later fractionated to yield the finished products. In the case of the Most (Brüx) phenosolvan extract, the low-reflux preliminary distillation was carried beyond the xylenol range. After the xylenols had been removed, distillation was continued with no reflux to yield crude pyrocatechol. The latter was then processed separately.

(b) A.S.W. and Brabag Crude Tar Acids: Frames 690-691.

These crude acids originated from CO_2 acidification of phenolates derived from Koppers steam-stripping dephenolization plants. They contained water and 2 to 5 percent of soda. Removal of the soda was necessary in order to prevent polymerization and coking in the stills and heaters. These crude acids, along with some others, were treated with sulfuric acid before distillation. A refining still, to be described later under "Fractional Distillation of Refined Tar Acids," was used for this purpose.

Forty metric tons of crude wet acids were charged to one of the refining stills, together with about 1,000 gallons of water, and mixed by recirculation with 75 or 96 percent sulfuric acid. Acidification was considered complete at Congo Red endpoint. The recirculating pump was stopped, and, after gravity separation, about 2,600 gallons of sodium sulfate solution was drawn off. The acidified tar acids remaining in the still were again recirculated and neutralized with just enough caustic soda to combine with the free fatty acids. Presence of the latter during distillation would cause severe corrosion. The yield of acidified acids was about 8,700 gallons with an ash content of approximately 0.5 percent. The treating time was about 12 hours, and approximately 2,000 metric tons per month of crude wet acids could be processed in one still.

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As a final step in this pretreatment of crude tar acids, the acidified acids were given a rough distillation through one of the bell-and-tray columns used for refining purposes. A charge of 50 metric tons yielded 33-40 metric tons of distilled tar acids, 2,100-2,600 gallons of water and forerunnings, and 4-10 metric tons of residue. The distillation time was 12 hours, and capacity per unit was approximately 2,000 metric tons a month of acidified tar acids. A small amount of reflux was used during dehydration but was stopped altogether when dry tar acids appeared. The end point of the distillation was determined by the viscosity of the residue at 50° C. (122° F.).

(c) Preliminary Distillation of Other Crude Tar Acids: Frames 687-688; 693.

Crude tar acids from the Leuna coal-hydrogenation plant and other sources, which did not have an excessive content of ash and high boiling portions, were predistilled without fractionation from a still having no column. Figure 38 (frame 693) shows the arrangement of two such stills.

The average composition of the still charge was as follows:

	Percent
Carbon residue	0.5
Water	15
Phenol	25
Cresols	35
Xylenols	10
High boilers and residue	14.5

The following materials were obtained:

	Percent (about)
Water and forerunnings	15
Distilled tar acids	67
Residue (alkyl phenols containing about 3 percent carbon residue)	18

The still of approximately 9,500-gallon capacity had no column. It was lined with acid-resisting material. A layer of Raschig rings at the top of the still prevented liquid spray carry-over to the condenser. Vacuum, down to 5 mm. (0.2 inch) Hg absolute, was applied from a five-stage steam-ejector assembly. The heating was provided by high-pressure steam in a vertical, 10-meter (32.8-foot), long-tube evaporator extending into the bottom of the still. An Eckardt meter measured the tar-acid charge to the still. This meter did not prove satisfactory for measuring residues after distillation. A reflux set-up was installed for better separation of water-tar acid forerunnings but did not prove successful.

Five thousand two hundred and eighty gallons (or 20 m^3) of the crude tar acids were charged from storage into the still. Vacuum was applied, and

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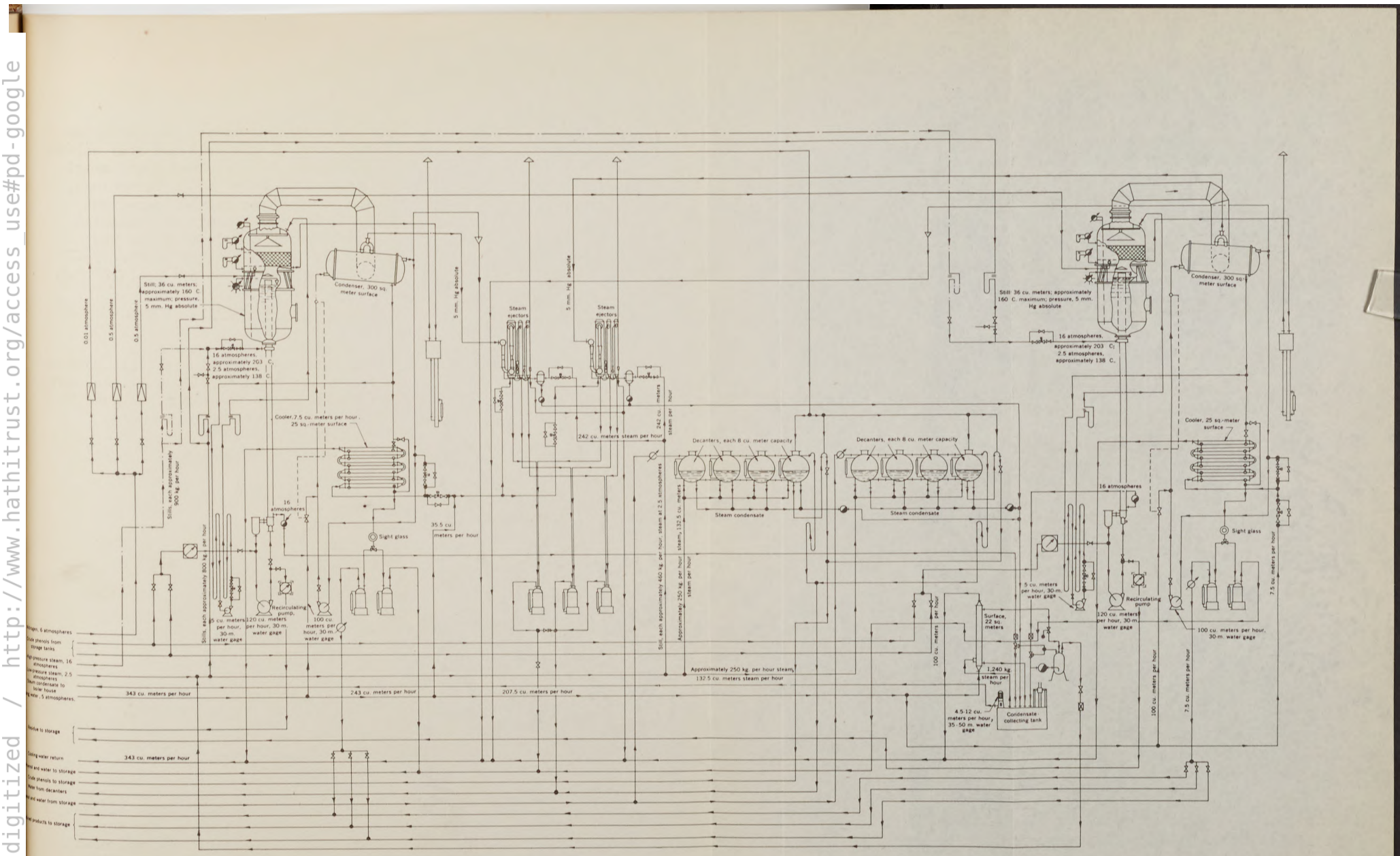
Still; 36 cu. meters; approximately 160° C. maximum; pressure, 5 mm. Hg absolute
Condenser, 300 sq. meter surface
16 atmospheres, approximately 203° C.
2.5 atmospheres, approximately 138° C.
Still; 36 cu. meters; approximately 160° C. maximum; pressure, 5 mm. Hg absolute
Condenser, 300 sq. meter surface
16 atmospheres, approximately 203° C.
2.5 atmospheres, approximately 138° C.
Steam ejectors
Steam ejectors
5 mm. Hg absolute
5 mm. Hg absolute
242 cu. meters steam per hour
Cooler, 7.5 cu. meters per hour, 25 sq.-meter surface
Cooler, 25 sq.-meter surface
Decanters, each 8 cu. meter capacity
Decanters, each 8 cu. meter capacity
Steam condensate
Steam condensate
16 atmospheres
16 atmospheres
Sight glass
Sight glass
35.5 cu. meters per hour
Still, each approximately 800 kg. per hour
Still, each approximately 460 kg. per hour, steam at 2.5 atmospheres
Approximately 250 kg. steam, 132.5 cu. meters steam per hour
Approximately 250 kg. per hour steam, 132.5 cu. meters steam per hour
7.5 cu. meters per hour, 30-m. water gage
Recirculating pump, 120 cu. meters per hour, 30-m. water gage
Recirculating pump, 120 cu. meters per hour, 30-m. water gage
45 cu. meters per hour, 30-m. water gage
100 cu. meters per hour, 30-m. water gage
100 cu. meters per hour, 30-m. water gage
Surface, 22 sq. meters
1,240 kg. steam per hour
4.5-12 cu. meters per hour, 35-50 m. water gage
Condensate collecting tank
100 cu. meters per hour
100 cu. meters per hour
7.5 cu. meters per hour
7.5 cu. meters per hour
343 cu. meters per hour
243 cu. meters per hour
207.5 cu. meters per hour
343 cu. meters per hour
0.01 atmosphere
0.5 atmosphere
0.5 atmosphere
2.5 atmosphere
2.5 atmosphere
Crude phenols from storage tanks
Low pressure steam, 2.5 atmospheres
Steam condensate to boiler house
Cooling water, 5 atmospheres
Residue to storage
Cooling water return
Crude phenols to storage
Water from decanters
Oil and water from storage
Heat products to storage

Figure 38. - Arrangement for preliminary distillation of tar acids at Leuna, I. G. Farbenindustrie, Merseburg.

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As a final step in this pretreatment of crude tar acids, the acidified acids were given a rough distillation through one of the bell-and-tray columns used for refining purposes. A charge of 50 metric tons yielded 33-40 metric tons of distilled tar acids, 2,100-2,600 gallons of water and forerunnings, and 4-10 metric tons of residue. The distillation time was 12 hours, and capacity per unit was approximately 2,000 metric tons a month of acidified tar acids. A small amount of reflux was used during dehydration but was stopped altogether when dry tar acids appeared. The end point of the distillation was determined by the viscosity of the residue at 50° C. (122° F.).

(c) Preliminary Distillation of Other Crude Tar Acids: Frames 687-688; 693.

Crude tar acids from the Leuna coal-hydrogenation plant and other sources, which did not have an excessive content of ash and high boiling portions, were predistilled without fractionation from a still having no column. Figure 38 (frame 693) shows the arrangement of two such stills.

The average composition of the still charge was as follows:

	Percent
Carbon residue	0.5
Water	15
Phenol	25
Cresols	35
Xylenols	10
High boilers and residue	14.5

The following materials were obtained:

	Percent (about)
Water and forerunnings	15
Distilled tar acids	67
Residue (alkyl phenols containing about 3 percent carbon residue)	18

The still of approximately 9,500-gallon capacity had no column. It was lined with acid-resisting material. A layer of Raschig rings at the top of the still prevented liquid spray carry-over to the condenser. Vacuum, down to 5 mm. (0.2 inch) Hg absolute, was applied from a five-stage steam-ejector assembly. The heating was provided by high-pressure steam in a vertical, 10-meter (32.8-foot), long-tube evaporator extending into the bottom of the still. An Eckardt meter measured the tar-acid charge to the still. This meter did not prove satisfactory for measuring residues after distillation. A reflux set-up was installed for better separation of water-tar acid forerunnings but did not prove successful.

Five thousand two hundred and eighty gallons (or 20 m^3) of the crude tar acids were charged from storage into the still. Vacuum was applied, and

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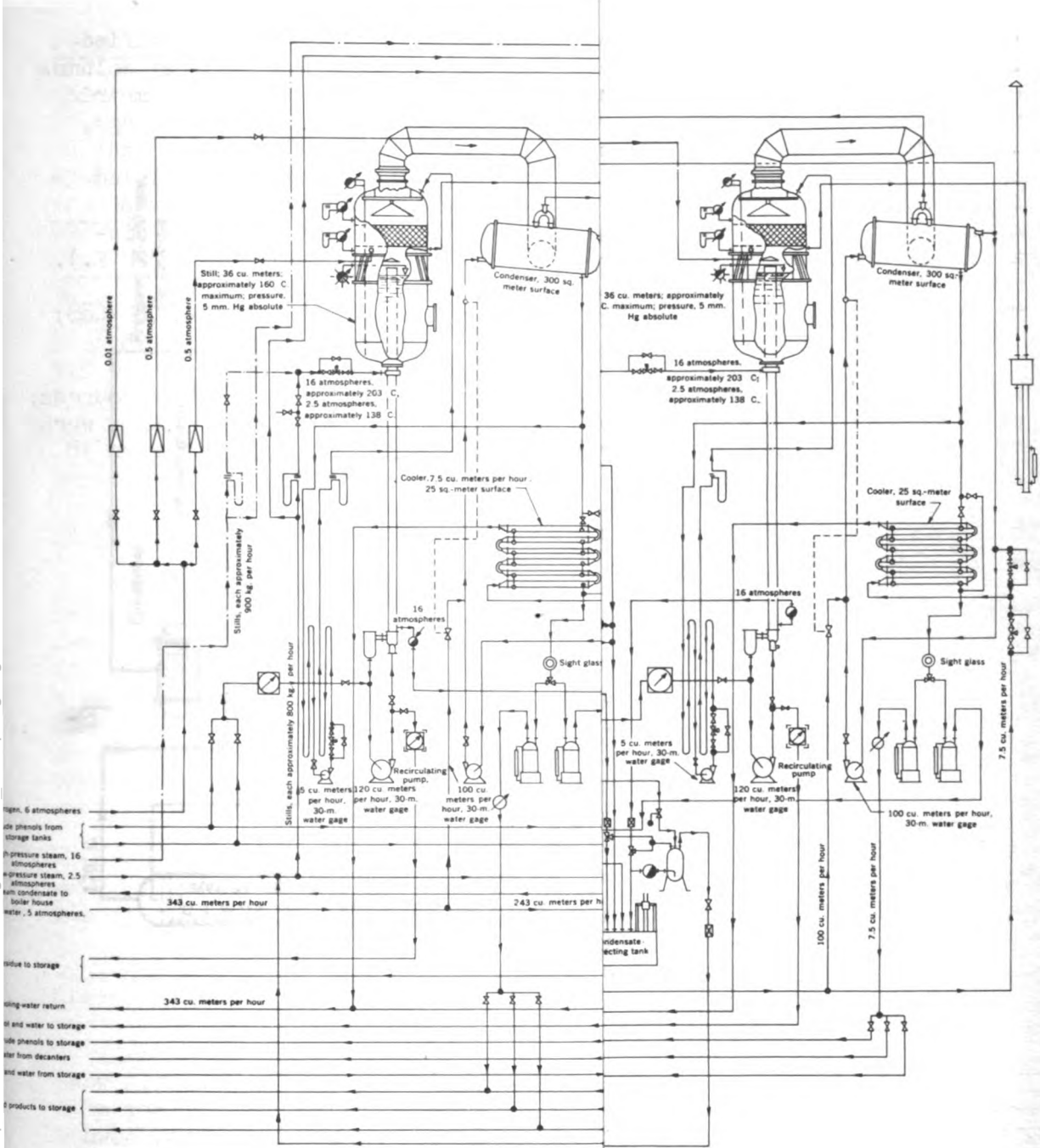
0.01 atmosphere
0.5 atmosphere
0.5 atmosphere
Still; 36 cu. meters; approximately 160 C. maximum; pressure, 5 mm. Hg absolute
Condenser, 300 sq. meter surface
16 atmospheres, approximately 203 C.; 2.5 atmospheres, approximately 138 C.
Cooler, 7.5 cu. meters per hour; 25 sq.-meter surface
Stills, each approximately 900 kg. per hour
16 atmospheres
Sight glass
Stills, each approximately 800 kg. per hour
5 cu. meters per hour, 30-m. water gage
Recirculating pump
120 cu. meters per hour, 30-m. water gage
100 cu. meters per hour, 30-m. water gage
343 cu. meters per hour
243 cu. meters per h
Condensate-collecting tank
36 cu. meters; approximately C. maximum; pressure, 5 mm. Hg absolute
16 atmospheres, approximately 203 C.; 2.5 atmospheres, approximately 138 C.
Condenser, 300 sq.-meter surface
Cooler, 25 sq.-meter surface
16 atmospheres
Sight glass
5 cu. meters per hour, 30-m. water gage
Recirculating pump
120 cu. meters per hour, 30-m. water gage
100 cu. meters per hour, 30-m. water gage
7.5 cu. meters per hour
100 cu. meters per hour
7.5 cu. meters per hour
gen, 6 atmospheres
cle phenols from storage tanks
h-pressure steam, 16 atmospheres
w-pressure steam, 2.5 atmospheres
am condensate to boiler house
water, 5 atmospheres
343 cu. meters per hour
sidue to storage
oling-water return
il and water to storage
ude phenols to storage
ater from decanters
and water from storage
products to storage

Figure 38. - Arrangement fo burg.

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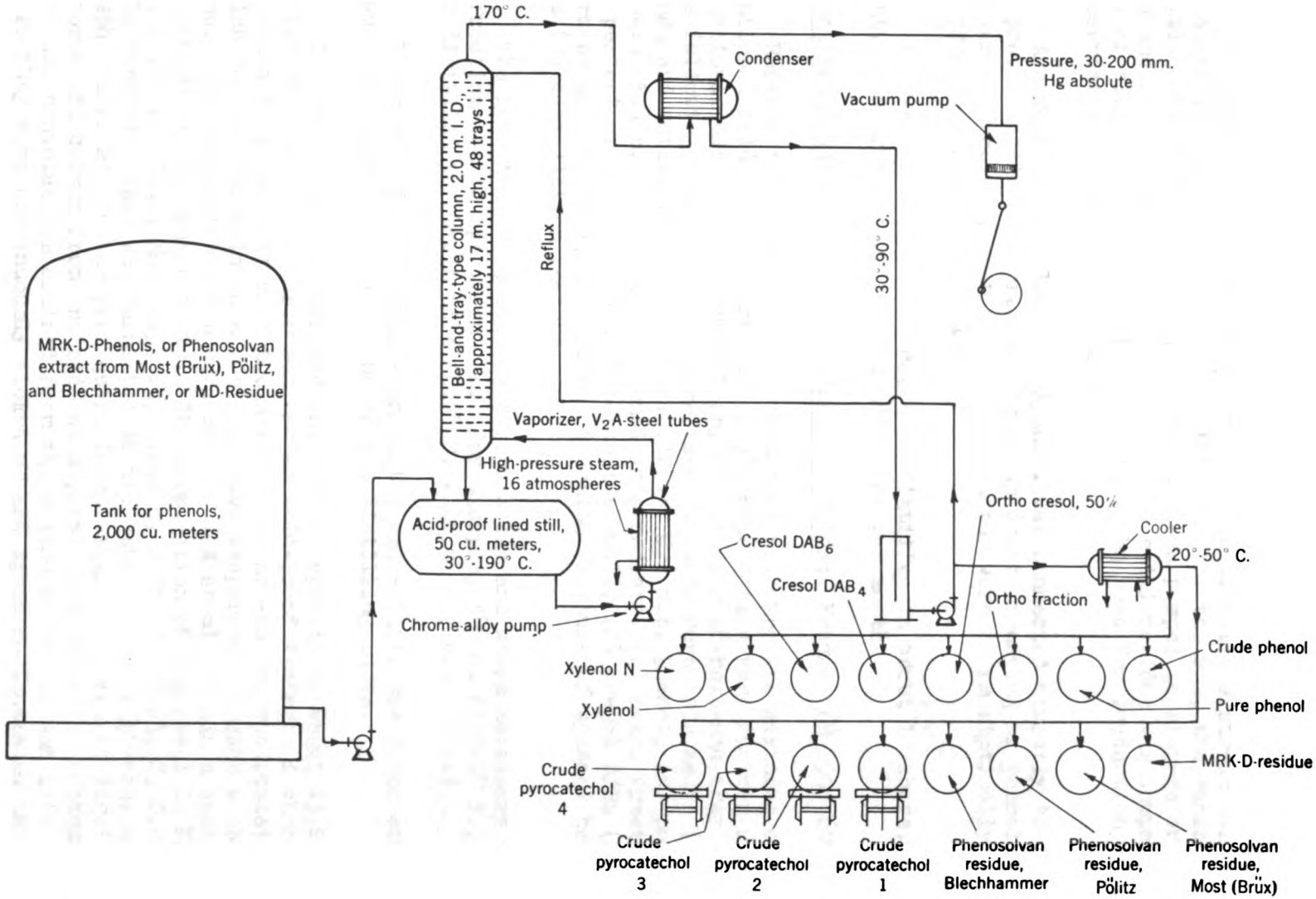

Figure 39. - Flow diagram for fractional distillation of refined tar acids at Leuna, I. G. Farbenindustrie, Merseburg.

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steam was turned into the evaporator. The maximum still temperature was 160° C. (320° F.). Condensed water and forerunnings flowed through a sight glass at about 20°-30° C. (68°-86° F.) to decanting tanks. When the liquid in the sight glass was no longer milky, the distillate stream was directed to distilled tar acids storage. At the end of the run, the vacuum was killed by bleeding in nitrogen gas, the residue was pumped to storage or tank car, and another run was started at once. The still residue was composed primarily of alkylated phenols. It was said to be used for production of a wood preservative known at "Basileum."

A safety trap filled with xylenol was connected directly to the still to prevent accidental pressure build-up due to freezing of phenol in the condenser and coolers, even though these later were thermostatically controlled at predetermined safe levels.

A 20-metric ton charge could be distilled in about 12 hours, or equivalent, to approximately 1,000 metric tons per month.

Fractional Distillation of Refined Tar Acids: Frames 688-690; 695; 696.

Refined tar acids prepared from various composite mixtures of the aforementioned acid-treating and distillation processes were further resolved by fractional distillation through any one of four 48-bell-and-tray columns. The following finished and semirefined products were obtained: (a) Forerunnings and water, (b) crude 38° C. (100.4° F.) m.p. phenol, (c) pure 40.2° C. (104.4° F.) m.p. phenol, (d) phenol intermediate, (e) ortho fraction (40 to 50 percent ortho cresol and 25 percent phenol), (f) cresol DAB4 (at least 38 percent meta cresol), (g) xylenol fraction (mixture of xylenol homologues), and (h) distillation residue.

The fractions were collected separately and where necessary were again refractionated to specification. For example, 38° C. (100.4° F.) m.p. phenol was distilled to obtain more 40.2° C. (104.4° F.) m.p. phenol.

There were four distillation units, and a fifth was under construction. Figure 39 (frame 695) shows one of the distilling units.

The still (each column had one) had a capacity of about 13,200 gallons. It was made of wrought iron but had an acid-proof lining. A chrome-steel pipe extended from the bottom of the still to a cast-chrome recirculating pump, which fed the tar acid to a vertical-tube evaporator through a chrome-steel line. The evaporator tubes were made of V2A steel and had a heating surface of 100 m^2 (1,076 square feet). The composition of V2A steel is Fe, 72.0; Cr, 20.3; Ni, 7.12; Mn, 0.23; Cu, 0.05; C, 0.25; Si, 0.23; S, 0.02; and P, 0.01 percent. Heat was furnished to the tube nest by high-pressure (16 atmospheres or 235 pounds per square inch) steam. The heated liquid and vapors entered the column from the side at a point just above the bottom. Condensate and nonvolatiles drained from the bottom of the column into the still, where they were continuously picked up and again recirculated through the heater.

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Vacuum to the distillation system was applied through a piston vacuum pump of about 600 cubic meters (21,190 cubic feet) per hour capacity. The working range during distillation was 200 to 30 mm. (about 7.9 to 1.2 inches) of Hg absolute.

The column was of the bell-and-tray type, 56 feet high, and had an inside diameter of 6.5 feet. The column had 48 cast-iron trays. All other parts of the column were of wrought iron.

Fifty-four and seven-tenths metric tons of refined tar acids were charged to the still from a 528,000-gallon storage tank. After charging, vacuum was applied, the recirculating pump was started, and heat was supplied to the evaporator. During distillation, the still temperature ranged from 30° to 190° C. (86°-374° F.). The exit vapors from the top of the column averaged about 120° C. (248° F.). They were condensed to 30° to 90° C. (86° to 194° F.), and reflux was pumped back to the top of the column at ratios of 2:1 to 9:1 during distillation.

To avoid excessive losses in the steam of the comparatively volatile phenol, the steam and vapors leaving the condonser were passed through a tower, where they were scrubbed with xylenol, which has a relatively low-steam volatility. The product going to receiving pans and storage was further cooled, 20°-50° C. (68°-122° F.), by a thermostatically controlled water cooler. Precaution was taken to prevent any sudden pressure build-up due to freezing of phenol while at the same time safeguarding the quality of the product by not holding it in storage at too high temperatures.

The pressure drop through the column under normal operating conditions was as high as 240 mm. (9.4 inches) of Hg.

A still charge required about 40 hours running time. At this rate, a single column could fractionate approximately 500 metric tons of refined tar acids in a month.

As in all of the other tar-acid distillations, the vacuum was released at the end of the run by drawing in nitrogen gas.

Sulfuric Acid-Refining of Crude Cresol: Frames 688; 690.

Crude cresol, DAB_4, as obtained by the fractional distillation of tar acids, was further purified by sulfuric-acid treatment and redistillation to yield a refined cresol DAB_4. The latter, containing at least 38 percent m-cresol, was a German pharmacopoeia specification. The acid treating and distillation took place in the bell-and-tray column assembly just described. The still was charged, as for a regular distillation run. Two percent by volume of concentrated sulfuric acid was charged from storage through the recirculating pump. The mixture was recirculated for intimate contact and then distilled through the column at 5 mm. (0.2 inch) of Hg vacuum absolute. At the end of the distillation the still residue temperature increased to a maximum of 110°-115° C. (230°-239° F.).

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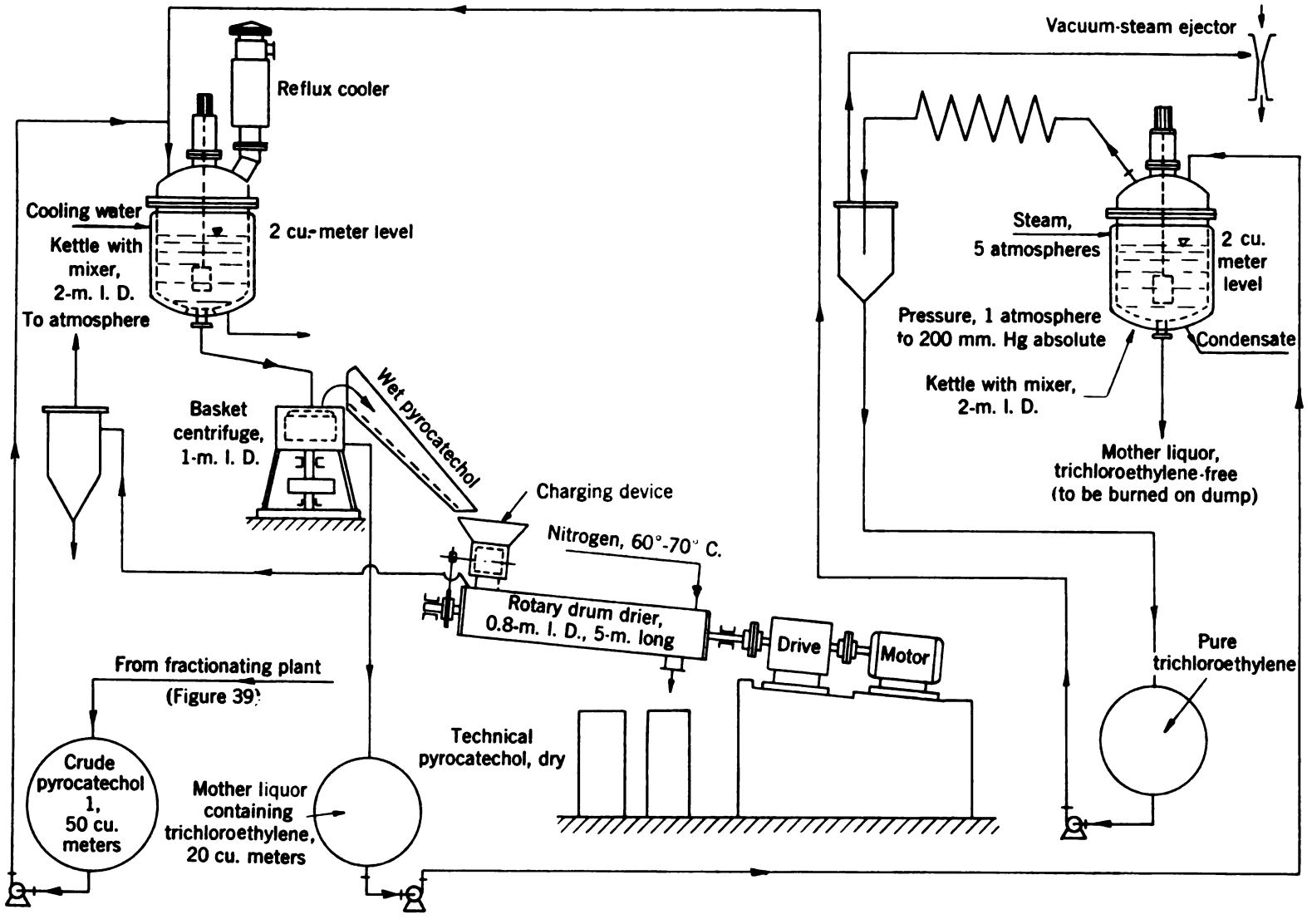

Figure 40. - Plant for production of pyrocatechol at Leuna, I. G. Farbenindustrie, Merseburg.

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The addition of sulfuric acid tied up the tar bases and had a resinifying effect upon the neutral unsaturated constituents of the crude cresols. A high vacuum had to be maintained throughout the distillation in order to prevent too much of an increase in the still temperature, with consequent decomposition of the tar-base sulfates. During the distillation, a small amount of aqueous forerunnings was obtained. The residue was about 10 percent of the charge. The dry distillate now met German pharmacopoeia specifications for refined cresol DAB4, which must contain at least 38 percent meta cresol. By further fractionation, cresol DAB6 could be produced that contained at least 50 percent meta cresol.

Production of Pyrocatechol: Frames 691-692; 697.

Pyrocatechol was produced in a plant that had been converted from pilot-plant operation. Figure 40 (frame 697) is a flow diagram of this plant.

As previously described, the residue from the Most (Brüx) phenosolvan extract distillation was further distilled to yield a crude pyrocatechol fraction. This material contained about 50 percent of pyrocatechol and about 30 percent of pyrocatechol homologues. The crude, which was stored in a 13,200-gallon tank, had a freezing point of 50°-55° C. (122°-131° F.).

Two hundred and sixty-four gallons of the melted crude was pumped at 60°-70° C. (140°-158° F.) into a kettle containing 264 gallons of trichloroethylene. The kettle was equipped with an agitator, cold-water jacket, and reflux condenser. After solution, the mixture was cooled and stirred until the temperature dropped to 20°-30° C. (68°-86° F.), at which point crystallization was considered complete.

The crystalline paste was centrifuged, washed with trichloroethylene while still spinning, and then dried in a rotating drum drier by means of 60°-70° C. (140°-158° F.) nitrogen gas. The exit nitrogen gas was passed through a dust filter to recover additional solids. Trichloroethylene carried over with the nitrogen was not recovered. After drying, the pyrocatechol was collected in paper bags. This product, known as technical-grade pyrocatechol, contained 95 percent of pyrocatechol plus 5 percent of pyrocatechol homologues. It had a freezing point of 102° C. (215.6° F.).

The mother liquor from the centrifuging operation was pumped into another reaction kettle, which was temporarily set up as a still. Here trichloroethylene was recovered by distillation. Toward the end of the distillation a vacuum of 200 mm. (about 7.9 inches) Hg absolute was applied in order to strip out the last traces of solvent. Yields from a 264-gallon batch of crude pyrocatechol were about 990 pounds of technical pyrocatechol and about 1,200 pounds of residue. This residue still contained about 20 percent pyrocatechol along with 50 percent of pyrocatechol homologues and was disposed of by burning. The working time for a 264-gallon charge was about 12 hours. Twenty metric tons of technical pyrocatechol could be produced in a month.

Extended research on the recovery, purification, and use of pyrocatechol has been reported by Dr. F. A. Croy - frames 1308-1369. Extensive research

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work on muconic acid derivatives from pyrocatechol was reported by Dr. G. Spengler - frames 718-738.

An important consideration in the production of pyrocatechol was the potential use of this compound in the leather-tanning industry, particularly in Czecho-Slovakia.

Hydrogenation of Phenol to Cyclohexanol: Frames 698-706.

The phenol used in the hydrogenation to cyclohexanol was the 40.2° C. (104.4° F.) m.p. material obtained from the tar-acid refining stills. It contained about 0.1-0.2 percent sulfur. On the basis of the phenol used, the conversion to cyclohexanol was about 99 percent.

The plant had 13 hydrogenation units, of which 7 were ready for operation. A single unit, theoretically, could produce about 90 metric tons of cyclohexanol per month, barring equipment failure and necessity for changing catalyst. Considering repairs and catalyst renewal, 500 metric tons of cyclohexanol per month production was estimated for the plant.

Essentially the hydrogenation process (fig. 41, frame 706), consisted of passing a mixture of phenol and hydrogen over a nickel-aluminum oxide catalyst at a temperature of about 150° C. (302° F.) and a pressure of 15-20 atmospheres (220-294 pounds per square inch). An exothermic reaction took place with considerable evolution of heat, which had to be adequately removed by external cooling. The reduction products were then cooled, and cyclohexanol was separated by distillation from unreacted phenol (which was returned to the system) and from small amounts of byproduct cyclohexanone and water.

The catalyst chamber was a steel cylinder (26 feet long and 2.6 feet in diameter) containing a nest of 211 tubes, each 1.3-inch in diameter, which were filled with the catalyst pellets.

The catalyst (No. Le 6523) was made at Leuna by adding sodium aluminate solution to a solution of nickel nitrate in water. The precipitated aluminum and nickel hydroxide mixture was washed, filtered, dried, pressed into pellets, and then reduced with hydrogen. Before reduction, the catalyst contained 1 mole part nickel hydroxide to 1 mole part aluminum hydroxide. The reduced mixture contained 30 to 40 percent nickel metal.

Highly purified hydrogen bubbled through a constant level layer of phenol (at 130°-145° C. or 206°-293° F.) at the bottom of the hydrogenation tower and became saturated with phenol. The vapor mixture then went through a layer of Raschig rings and then through the catalyst chamber, where the hydrogenation took place.

The process was completely continuous and automatic. Under good conditions, such as a very low-sulfur content in the phenol, the catalyst lasted for about 3 months before renewal was necessary. By this time, tarry and high-sulfur-containing matter had accumulated in the evaporator at the bottom of the hydrogenation tower. The quantity was about 25 gallons and was discarded.

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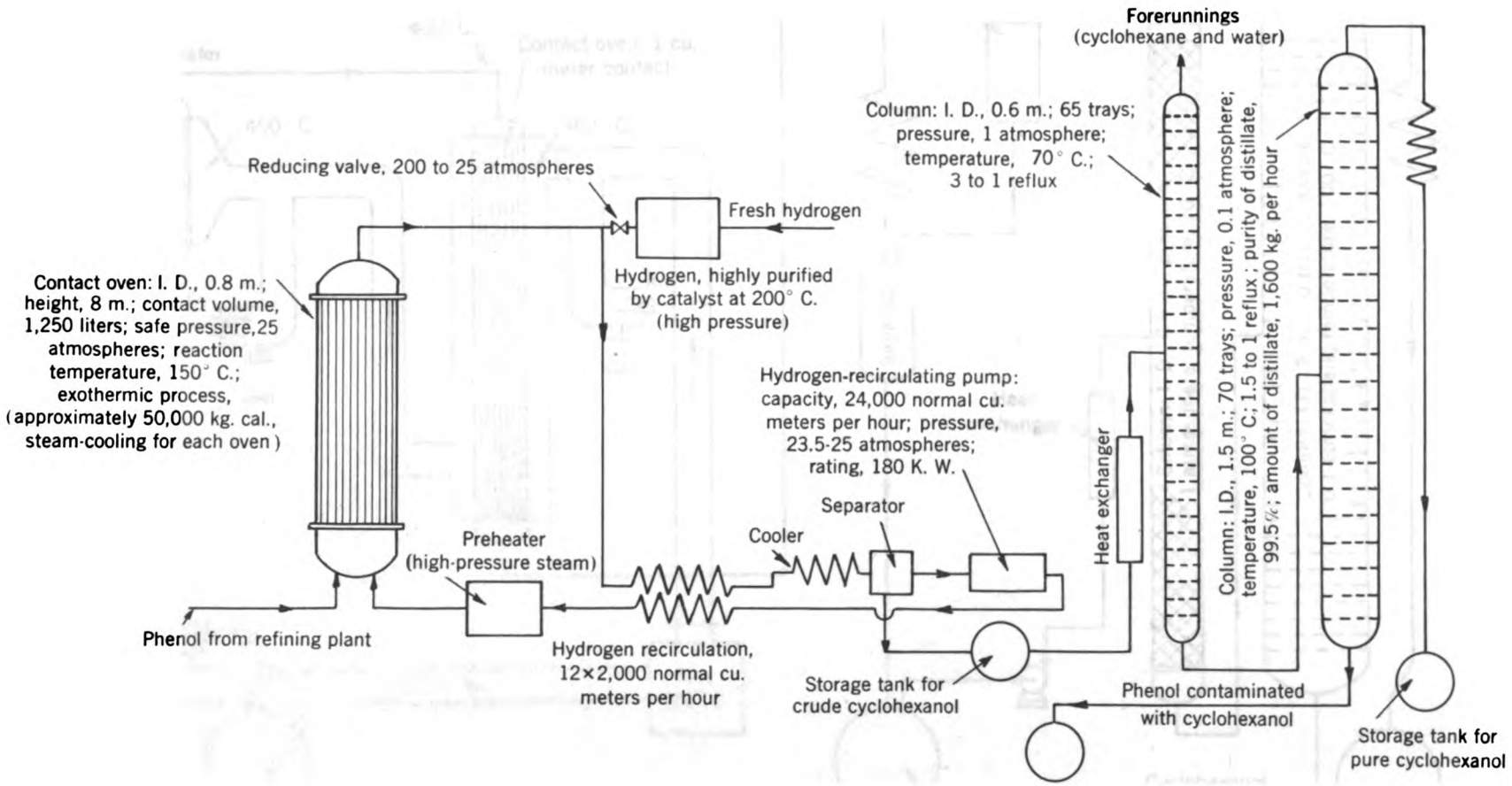

Figure 41. - Arrangement for hydrogenation of phenol to cyclohexanol at Leuna, I. G. Farbenindustrie, Merseburg.

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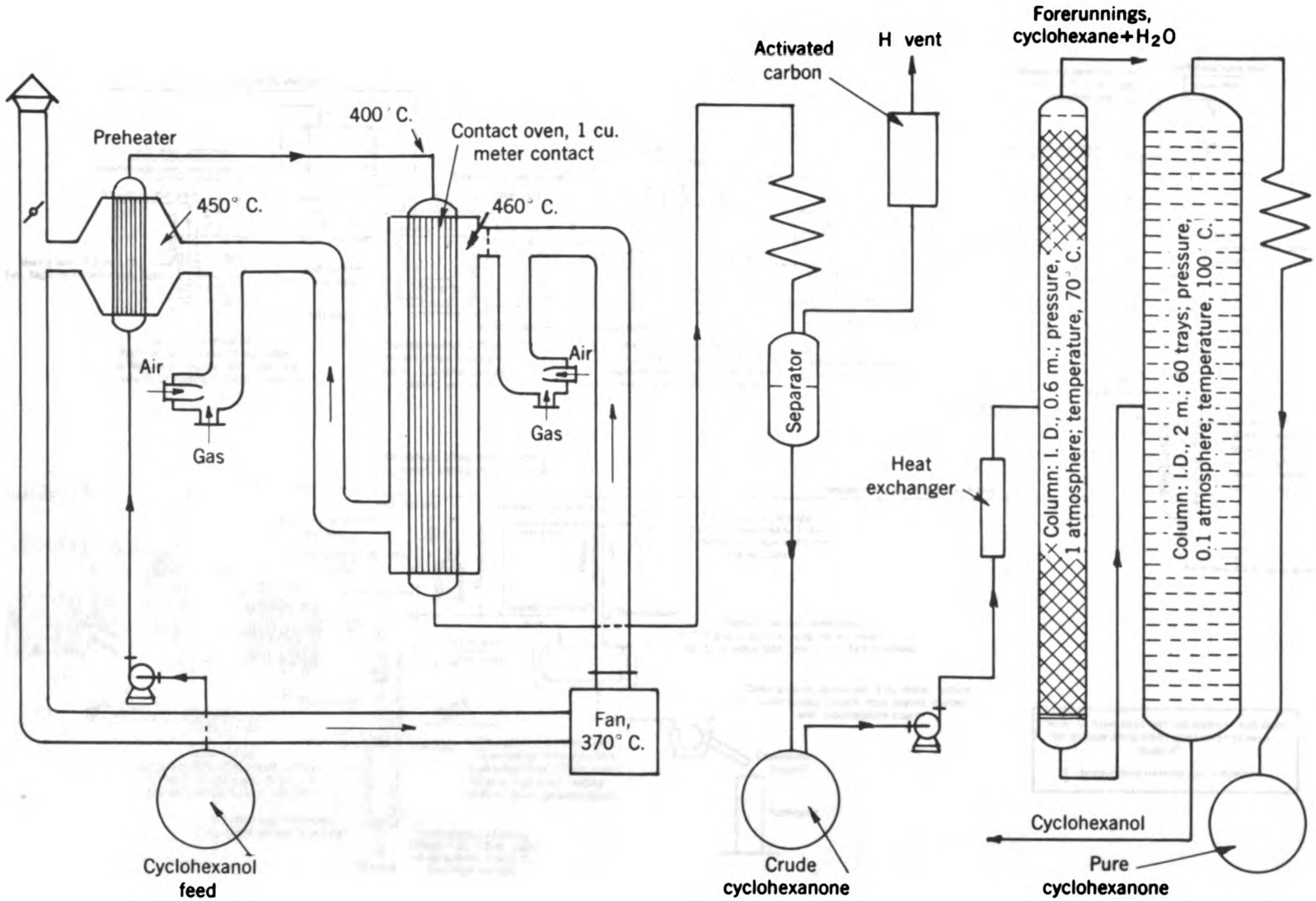

Figure 42. - Flow diagram for production of cyclohexanone at Leuna, I. G. Farbenindustrie, Merseburg.

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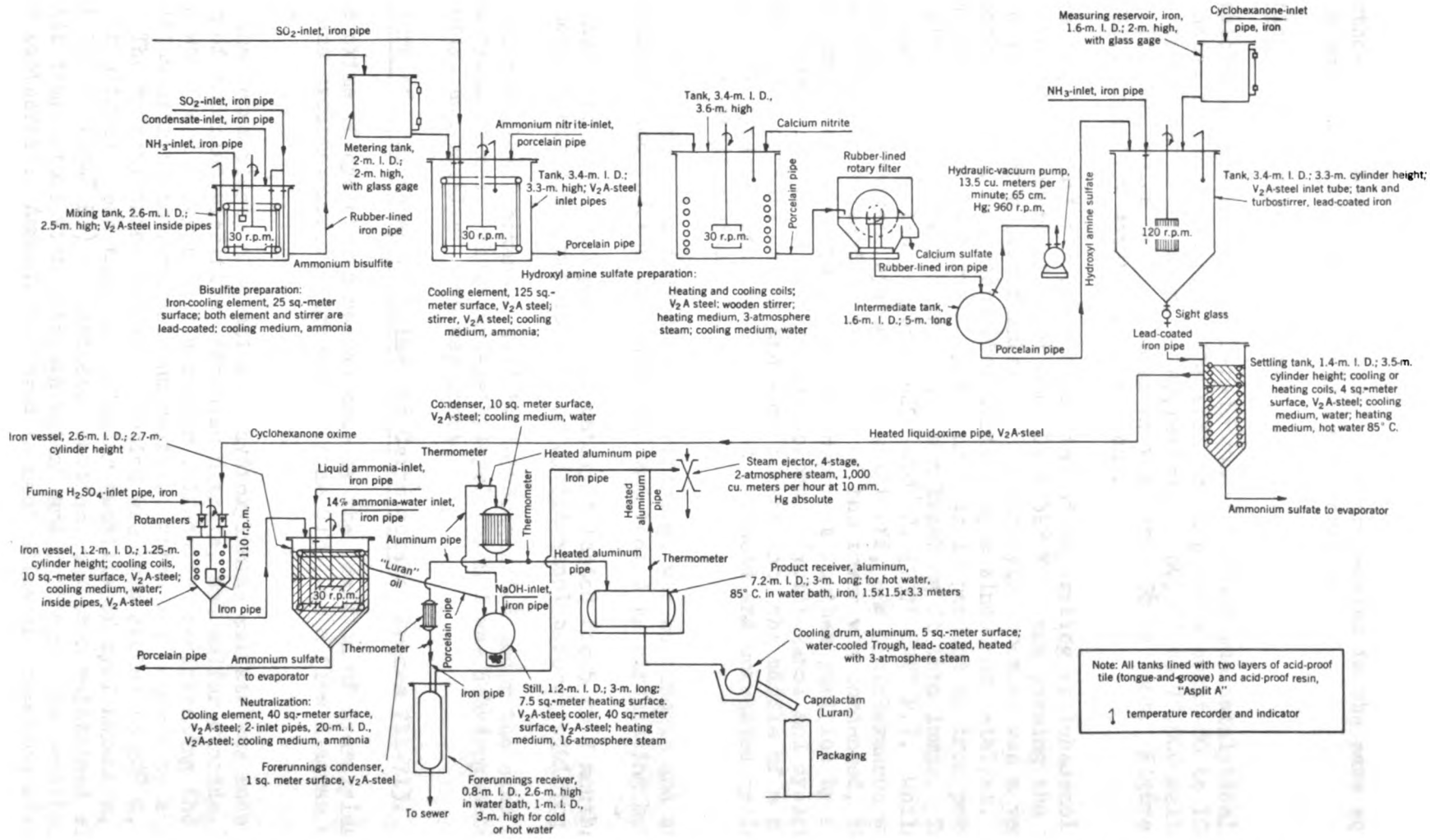
SO2-inlet, iron pipe
SO2-inlet, iron pipe
Condensate-inlet, iron pipe
NH3-inlet, iron pipe
Mixing tank, 2.6-m. I. D.; 2.5-m. high; V2A-steel inside pipes
30 r.p.m.
Rubber-lined iron pipe
Ammonium bisulfite
Bisulfite preparation: Iron-cooling element, 25 sq.-meter surface; both element and stirrer are lead-coated; cooling medium, ammonia
Metering tank, 2-m. I. D.; 2-m. high, with glass gage
Ammonium nitrite-inlet, porcelain pipe
Tank, 3.4-m. I. D.; 3.3-m. high; V2A-steel inlet pipes
30 r.p.m.
Porcelain pipe
Cooling element, 125 sq.-meter surface, V2A steel; stirrer, V2A steel; cooling medium, ammonia;
Hydroxyl amine sulfate preparation:
Tank, 3.4-m. I. D., 3.6-m. high
Calcium nitrite
30 r.p.m.
Porcelain pipe
Heating and cooling coils; V2A steel; wooden stirrer; heating medium, 3-atmosphere steam; cooling medium, water
Rubber-lined rotary filter
Calcium sulfate
Rubber-lined iron pipe
Intermediate tank, 1.6-m. I. D.; 5-m. long
Porcelain pipe
Hydraulic-vacuum pump, 13.5 cu. meters per minute; 65 cm. Hg; 960 r.p.m.
Hydroxyl amine sulfate
Measuring reservoir, iron, 1.6-m. I. D.; 2-m. high, with glass gage
Cyclohexanone-inlet pipe, iron
NH3-inlet, iron pipe
Tank, 3.4-m. I. D.; 3.3-m. cylinder height; V2A-steel inlet tube; tank and turbostirrer, lead-coated iron
120 r.p.m.
Sight glass
Lead-coated iron pipe
Settling tank, 1.4-m. I. D.; 3.5-m. cylinder height; cooling or heating coils, 4 sq.-meter surface, V2A-steel; cooling medium, water; heating medium, hot water 85° C.
Ammonium sulfate to evaporator
Heated liquid-oxime pipe, V2A-steel
Condenser, 10 sq. meter surface, V2A-steel; cooling medium, water
Iron vessel, 2.6-m. I. D.; 2.7-m. cylinder height
Cyclohexanone oxime
Thermometer
Heated aluminum pipe
Liquid ammonia-inlet, iron pipe
Iron pipe
Heated aluminum pipe
Steam ejector, 4-stage, 2-atmosphere steam, 1,000 cu. meters per hour at 10 mm. Hg absolute
Fuming H2SO4-inlet pipe, iron
Rotameters
14% ammonia-water inlet, iron pipe
Thermometer
Aluminum pipe
Iron vessel, 1.2-m. I. D.; 1.25-m. cylinder height; cooling coils, 10 sq.-meter surface, V2A-steel; cooling medium, water; inside pipes, V2A-steel
110 r.p.m.
30 r.p.m.
"Luran" oil
Iron pipe
Heated aluminum pipe
Thermometer
Product receiver, aluminum, 7.2-m. I. D.; 3-m. long; for hot water, 85° C. in water bath, iron, 1.5×1.5×3.3 meters
NaOH-inlet, iron pipe
Porcelain pipe
Thermometer
Porcelain pipe
Ammonium sulfate to evaporator
Cooling drum, aluminum, 5 sq.-meter surface; water-cooled Trough, lead-coated, heated with 3-atmosphere steam
Caprolactam (Luran)
Packaging
Iron pipe
Still, 1.2-m. I. D.; 3-m. long; 7.5 sq.-meter heating surface. V2A-steel; cooler, 40 sq.-meter surface, V2A-steel; heating medium, 16-atmosphere steam
Neutralization: Cooling element, 40 sq.-meter surface, V2A-steel; 2 inlet pipes, 20-m. I. D., V2A-steel; cooling medium, ammonia
Forerunnings condenser, 1 sq. meter surface, V2A-steel
To sewer
Forerunnings receiver, 0.8-m. I. D., 2.6-m. high in water bath, 1-m. I. D., 3-m. high for cold or hot water
Note: All tanks lined with two layers of acid-proof tile (tongue-and-groove) and acid-proof resin, "Asplit A"
temperature recorder and indicator

Figure 43. - Flow diagram for manufacture of cyclohexanone oxime and caprolactam at Leuna, I. G. Farbenindustrie, Merseburg.

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Ortho- and meta-para cresol could be hydrogenated in the same equipment, but with somewhat different catalytic efficiencies.

Production of Cyclohexanone: Frames 707-710.

Cyclohexanol from the phenol-hydrogenation plant was catalytically dehydrogenated to cyclohexanone. The unit could process about 80 to 100 gallons of cyclohexanol per hour, or approximately 60,000 to 70,000 gallons per month. The conversion to cyclohexanone was about 98 percent. Figure 42 (frame 710) is a flow diagram of the plant.

The dehydrogenation process consisted of vaporizing cyclohexanol, preheating to a temperature of about 400° C. (752° F.), and passing the vapor into the top of a catalytic chamber. This catalytic chamber was a vertical tower packed with 37 two-inch tubes containing a zinc-iron catalyst. The catalyst was made at Bitterfeld by adding 8 to 12 percent of iron powder to molten zinc, casting the mixture, and then breaking it into lumps. The reaction chamber was heated to about 425°-450° C. (797°-842° F.). While passing over the heated catalyst, hydrogen was split off, and cyclohexanone was formed. The reaction product leaving the bottom of the tower was condensed, the hydrogen was vented, and the crude cyclohexanone was further purified by flashing off cyclohexane and water in a packed column. Cyclohexanol and cyclohexanone left the bottom of this column and were pumped into the middle of a plate column, where cyclohexanone was distilled overhead and unreacted cyclohexanol came out as a still residue.

The entire process, including the refining, was continuous and automatic. The evaporator and reactor were jacketed and heated by circulating hot air.

A charge of about 900 pounds of catalyst lasted for 5 to 7 months and dehydrogenated about 1,500 metric tons of cyclohexanol before requiring renewal.

Ortho-methylcyclohexanol could be dehydrogenated under the same conditions as cyclohexanol. Meta-para-methylcyclohexanol was dehydrogenated less efficiently, and catalyst life was short.

Manufacture of Cyclohexanone Oxime and Caprolactam: Frames 711-713.

The oxime of cyclohexanone was made by the reaction of hydroxylamine sulfate and cyclohexanone. Figure 43 (frame 713), is a flow diagram of the process.

In the Leuna plant, a solution of hydroxylamine sulfate was made by the reaction of ammonium bisulfite, ammonium nitrite, and sulfur dioxide. This solution was freed of byproduct ammonium sulfate by precipitating the latter with calcium nitrite to form calcium sulfate, which was removed by a rotary filter. The hydroxylamine sulfate solution was then cooled to 20° C. (68° F.), mixed with slightly less than an equimolar quantity of cyclohexanone, heated to about 90° C. (194° F.) to initiate reaction, and then maintained slightly below this temperature with thorough mixing and cooling. The reaction was strongly exothermic. Ammonia was used to neutralize the reaction mixture,

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which was then separated by decanting. Ammonium sulfate solution was drawn off as a bottom layer. The oily oxime formed at the top contained about 4 percent water.

This oxime next underwent a rearrangement with fuming sulfuric acid to form caprolactam or "luran." The oxime and fuming sulfuric acid, containing 25 percent free SO_3, were metered in a 1 to 1 ratio by weight and mixed at about 85° C. (185° F.) in a reaction vessel provided with strong agitation and cooling. This vessel had already been partly charged with the rearranged product (caprolactam) as diluent to reduce the extremely high exothermic reaction between the oxime and fuming sulfuric acid. After the rearrangement, the reaction mixture with a sulfuric-acid content of 55 percent was discharged continuously into a neutralization vessel, where it was neutralized with 14 percent ammonia water, care being taken to keep the temperature below 40° C. (104° F.). After settling, the oily lactam, containing about 30 percent of water, was decanted, vacuum dehydrated, and then distilled at about 10 mm. (0.4 inch) Hg vacuum absolute and 120° C. (248° F.). The heavier saturated ammonium sulfate solution resulting from the neutralization was drawn off from the bottom of the neutralization vessel.

This lactam was the raw material for the preparation of Igamid B, which was made at other plants of I. G. Farbenindustrie.

Manufacture of Adipic Acid: Frames 714-717.

Adipic acid was manufactured by nitric acid oxidation of cyclohexanol in V_2A steel equipment in accordance with the procedure illustrated in figure 44 (frame 716). The composition of V_2A steel was given on page 47.

Cyclohexanol and 62 percent nitric acid were added simultaneously to a reaction kettle with cooling and agitation. The reaction temperature was kept below 65° C. (149° F.). Evolved nitrogen oxides and carbon dioxide were absorbed in lime slurry. The oxidized mixture was air-blown in towers, and the oxide-free liquor was crystallized at 10°-15° C. (50°-59° F.). Adipic acid was separated by a centrifuge or ceramic filter and washed with mother liquor from the recrystallization step. The washed crystals were dissolved in distilled water at 85° C. (185° F.). Purified adipic acid was dried in a hot-air, rotary-drum drier, further dried and cooled with air in a second drum drier, and then packed in bags.

One hundred kilograms (220 pounds) of refined adipic acid required 80 kilograms (176 pounds) of cyclohexanol, 132 kilograms (291 pounds) of nitric acid (calculated as 100 percent), and 15 kilograms (33 pounds) of calcium oxide. Before destruction, the plant had a monthly capacity of 120 metric tons of adipic acid.

The mother liquor from crude adipic acid crystallization was concentrated for reuse to 62 percent nitric acid strength, either with solution of nitrogen oxides under pressure or by vacuum evaporation of excess water. Occasionally a part of this liquor was drawn off for recovery of the byproducts - succinic, glutaric, and oxalic acids.

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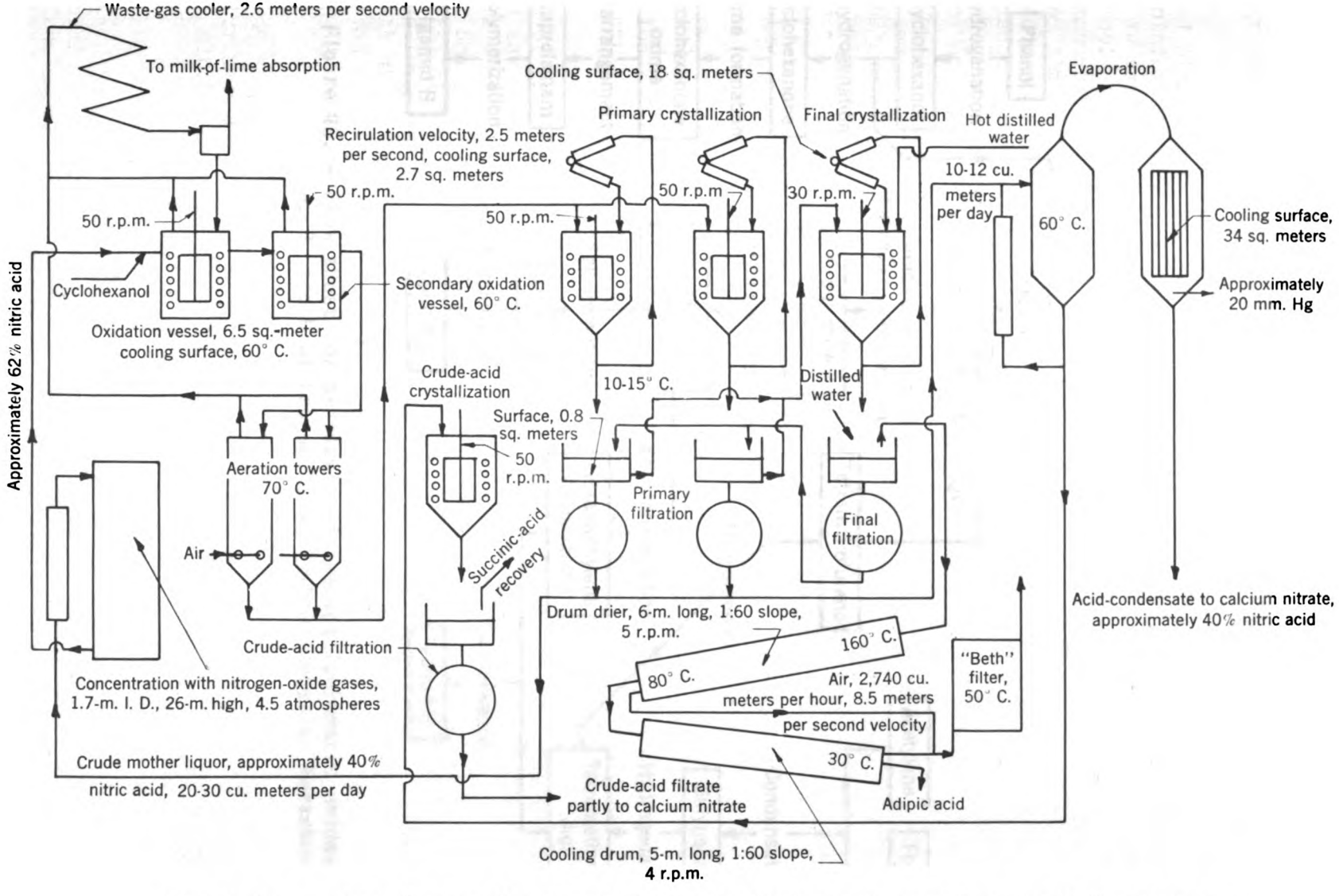
Waste-gas cooler, 2.6 meters per second velocity
To milk-of-lime absorption
Cooling surface, 18 sq. meters
Primary crystallization
Final crystallization
Evaporation
Hot distilled water
Recirulation velocity, 2.5 meters per second, cooling surface, 2.7 sq. meters
50 r.p.m.
50 r.p.m.
50 r.p.m.
50 r.p.m.
30 r.p.m.
10-12 cu. meters per day
60° C.
Cooling surface, 34 sq. meters
Approximately 20 mm. Hg
Approximately 62% nitric acid
Cyclohexanol
Secondary oxidation vessel, 60° C.
Oxidation vessel, 6.5 sq.-meter cooling surface, 60° C.
Crude-acid crystallization
10-15° C.
Distilled water
Surface, 0.8 sq. meters
50 r.p.m.
Aeration towers 70° C.
Air
Primary filtration
Final filtration
Succinic-acid recovery
Drum drier, 6-m. long, 1:60 slope, 5 r.p.m.
160° C.
80° C.
Air, 2,740 cu. meters per hour, 8.5 meters per second velocity
"Beth" filter, 50° C.
30° C.
Adipic acid
Acid-condensate to calcium nitrate, approximately 40% nitric acid
Crude-acid filtration
Concentration with nitrogen-oxide gases, 1.7-m. I. D., 26-m. high, 4.5 atmospheres
Crude mother liquor, approximately 40% nitric acid, 20-30 cu. meters per day
Crude-acid filtrate partly to calcium nitrate
Cooling drum, 5-m. long, 1:60 slope, 4 r.p.m.

Figure 44. - Flow diagram for manufacture of adipic acid, as of early 1944, at Leuna, I. G. Farbenindustrie, Merseburg.

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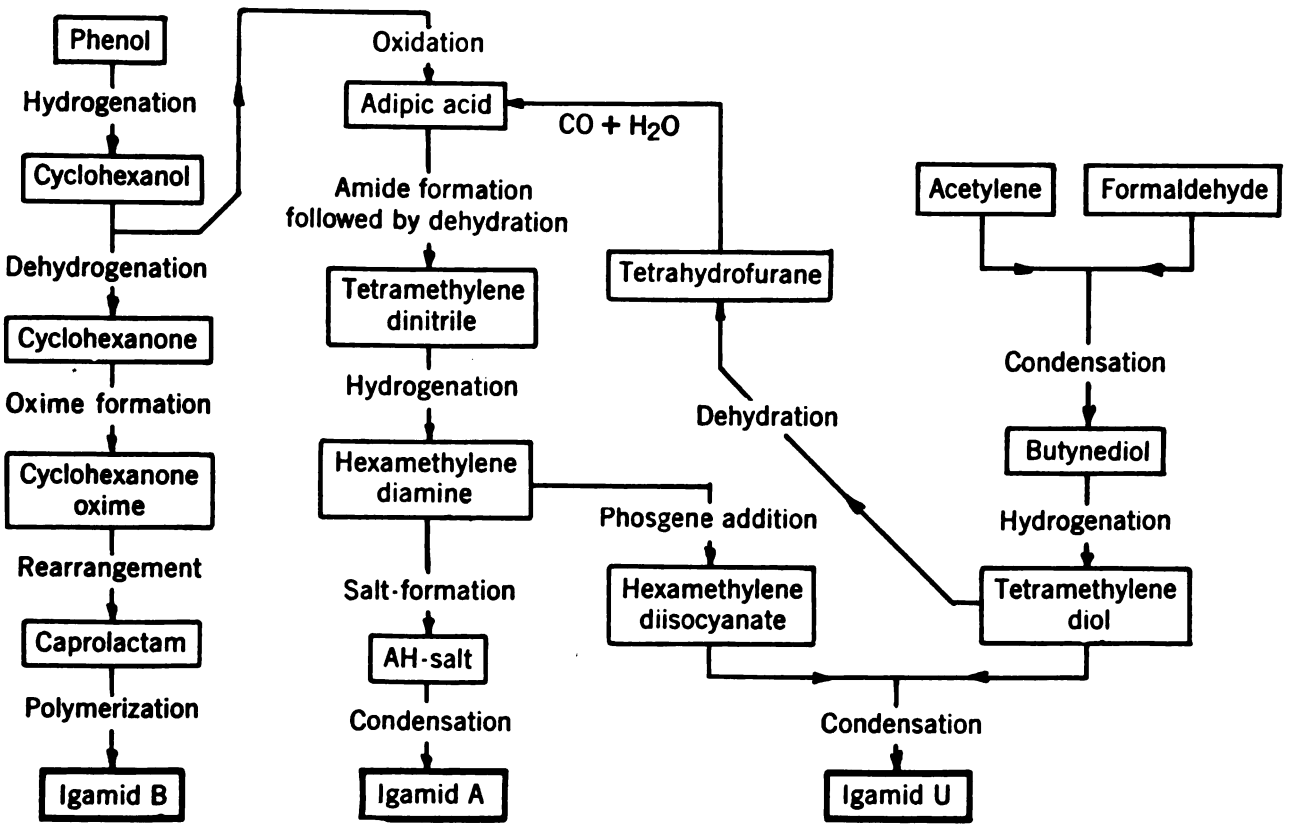

Figure 45. - Flow sheet for synthesis of Igamids (polymeric amides) A, B, and U at Leuna, I. G. Farbenindustrie, Merseburg.

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Manufacture of Igamids: Frame 717.

The various processes described in the foregoing pages, starting with the hydrogenation of phenol and ending with the manufacture of adipic acid, were all stages in the manufacture of polymeric amides, called Igamids. Their relationship to one another and to Igamids B, A, and U are illustrated in figure 45 (frame 717).

Caprolactam, made at Leuna with phenol as the starting material, was polymerized to Igamid B at Wolfen, Aceta, Lichtenberg, and Lansberg. The polymerization was reported in C. I. O. S. file XXVI-53, item 22 (PB 530), "Manufacture of Caprolactam, I. G. Farbenindustrie Leuna Werke, Merseburg", 1945, by R. H. Boundy and R. L. Hasche, to have been effected by heating the caprolactam under pressure in the presence of carbon dioxide.

Adipic acid, according to the accompanying flow sheet (fig. 45), was made by the oxidation of cyclohexanol. The adipic acid plant at Leuna, having a capacity of 120 metric tons per month, was destroyed during the war. A 40-50 metric ton per month plant was being constructed in January 1946, and the construction of a 550-metric ton per month plant was planned.

Tetramethylene dinitrile and hexamethylene diamine were made at Leuna during the war in pilot plants. The capacity of the former was about 30 metric tons per month. Both of these plants were destroyed. In January 1946, new pilot plants of about 10 metric tons per month capacity each were being constructed.

There was no plant at Leuna for making AH-salt, but a plant with a capacity of 10-20 metric tons per month was planned. Also, plans had been made for a plant to produce Igamid A at Wolfen Film.

The manufacture of Igamid U by condensing 1:6 hexamethylene diisocyanate with tetramethylenediol has been described in detail in C. I. O. S. file XXII-16, item 22 (PB 185), "I. G. Farbenindustrie A. G. Elberfeld & Leverkusen", 1945, by Francis J. Curtis. Also, the preparation of hexamethylene diisocyanate by phosgene addition to hexamethylene diamine, indicated in figure 44, is described in the same report by Curtis. It was stated at Leuna in January 1946 that a 20-30 metric ton per month plant for manufacturing Igamid U at Leverkeusen was planned.

Curtis, in the C. I. O. S. report mentioned above, described Igamid U as follows: "Igamid U does not combine with ordinary commercial plasticizers but can be injection and compression molded. The melting point is 184° C. (363.2° F.), with a short period between melting and low viscosity. Products have good mechanical strength and electrical properties and high resistance to solvents and oils. Fibers are produced by hot melt, spinning, and stretching. Bases for leather are made by shooting. Used for bristles (Perlon U), it has a lower melting point than Nylon, and therefore is easier to form, has a better resistance against acids, is harder, has better electrical properties, and has a maximum water absorption of 2 percent, compared to 8 percent for Nylon. Molecular weight - 10,000.".

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The various products shown in figure 44 that entered into the manufacture of tetramethylene diol and tetrahydrofurane, i.e., acetylene, formaldehyde, and butynediol, were all produced at Schkopau. The production of adipic acid at Schkopau from tetrahydrofurane was planned.

As indicated by figure 44, the principal purpose of the various processes mentioned above was to manufacture Igamids A, B, and U. Igamids were used in Germany for making leather substitutes, glues, injection molded products, textile materials, bristles, film, and various other commercial materials.

PART II. - BRIEF REPORTS ON VISITS TO GERMAN ORGANIZATIONS CONCERNED WITH THE PRODUCTION, PROCESSING, AND USE OF LOW-TEMPERATURE TAR

Lurgi - Frankfurt am Main

The Lurgi offices in Frankfurt am Main were visited by the writer on September 10 and 11, 1945. Lurgi was occupying the building across the street from Lurgihaus, 17-19 Gervinus Strasse. The Lurgi organization has three divisions:

Lurgi Gesellschaft für Wärmetechnik m.b.H., deals principally with matters pertaining to fuel and steam.

Lurgi Apparatebau Gesellschaft, builds electrical precipitation plants.

Lurgi Gesellschaft für Chemie and Hüttenwesen, builds principally sulfuric acid plants, metallurgical furnaces, etc.

On both visits, the director, Dr. Ing. F. A. Oetken, was interviewed concerning the production, processing, and use of low-temperature tar in Germany. Extraction of phenols from waste liquors and the extraction of paraffins from brown coal also were discussed briefly. A summary of the conversations about these matters follows:

The Krupp-Lurgi process is the only one used in Germany for the low-temperature carbonization of caking bituminous coal. Plants of this kind are situated at Wanne-Eickel and Velsen. Brennstoff Technik G.m.b.H. at Essen had plans for a steel furnace installation for this purpose with a capacity of 30,000 metric tons per year, but they were not carried out because of the war.

The only plants for the low-temperature carbonization of noncaking bituminous coal are of the Lurgi-Spülgas type. They are at Beuthen, Auschwitz, and Blechhammer. Plans were under consideration for this purpose by Didier-Werke A. G., Berlin, (25,000 metric tons per year), by H. Koppers, Essen (ceramic furnaces), and by Dr. C. Otto and Company, Bochum, but plants were not installed because of the war.

For soft brown coal, most of the plants are of the Lurgi-Spülgas type. The equipment is virtually the same as that used for noncaking bituminous coal. Such plants are situated at Offleben, Nachterstedt, Deuben, Regis,

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Deutzen, Espenhain, Böhlen, Hirschfelde, and Profen. Except for the plant at Offleben (near Helmstedt), all of these plants are in the Russian zone. A plant for soft brown coal, of the Borsig-Geissen type, is situated at Kulkwitz. Older types of equipment used in other plants are principally of the Rolle and old Geissen types.

Hard brown coal is carbonized in one Lurgi-Spülgas plant of 25,000 to 30,000 metric tons per day capacity at Most (Brüx).

The quantities of low-temperature tar produced in these various plants and the uses for the tar are summarized in table 1 (see page 12) and in table 6, both of which were prepared for the writer by Dr. Oetken. Referring to the data given in these tables, Dr. Oetken pointed out that of the 2.6 million metric tons of low-temperature tar made in Germany, only about 10 percent comes from bituminous coal and 90 percent comes from brown coal. Concerning the uses of low-temperature tar, Dr. Oetken stated that 88 percent of the tar from bituminous coal is hydrogenated. The amount of brown-coal tar that is hydrogenated is about 85 percent of the total. The low-temperature tar from bituminous coal that is not hydrogenated is processed by Gesellschaft für Teerverwertung. That from soft brown coal which is not hydrogenated is for the most part distilled by Riebeckschc Montanwerke, Anhaltische Kohlenwerke, Deutsche Erdöl A. G., and K. V. G. to obtain fuel oil, some petrol, some Diesel oil, paraffin wax, and a certain amount of coke for electrodes.

TABLE 6. - Treatment of low-temperature tar (compare with table 1)

From group No.	Method of use	Tons per year (metric)
I	Added to tar fuel oil by Gesellschaft für Teerverwertung, Duisburg-Meiderich	28,000
II	Hydrogenated	205,000
III	(a) Distillation with recovery of paraffin wax:	
	Riebeckschc Montanwerke, Webau	
	Anhaltische Kohlenwerke, Köpsen	1/400,000
	Deutsche Erdöl A. G., Rositz	
	K. V. G., Gölzau	
	(b) Distillation with Edeleanu extraction of paraffin wax:	
	Akt. Sächsische Werke, Espenhain	1/200,000
	(c) Hydrogenated	1/800,000
IV	Hydrogenated by Sudetenländische Treibstoffwerke, Most (Brüx)	1,000,000
		2,633,000

1/ About.

One new development of considerable importance is the extraction of soft brown-coal tar by the Edeleanu process (using SO_2) to obtain paraffin wax, then by distillation of the extracted material to obtain Diesel oil, fuel oil, and coke for electric furnaces. The process was developed by Aktiengesellschaft Sächsische Werke, Dresden, and the plant is at Espenhain. According

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to Dr. Oetken, the process is very successful, and working costs are much cheaper than those of the older process.

Lurgi's phenosolvan treatment of waste liquors from low-temperature carbonization plants consists in extracting the phenols with a solvent consisting principally of isobutyl acetate, distillation to recover the extracting medium, and further treatment of the liquor to remove traces of phenols. Plants of this kind are located at Most (Brüx) and Blechhammer. Liquor from hard brown coal contains up to 25 grams of phenols per liter (about 0.2 pound per U. S. gallon), and liquor from soft brown coal somewhat less.

Lurgi has developed a process for briquetting brown-coal dust under high pressure (2,200-2,500 kg. per sq. cm. or 31,290-35,560 lb. per sq. in.). Equipment for this purpose is constructed by Krupp-Grusonwerk at Magdeburg.

Lurgi had the agency for Söderberg electrodes in Germany but is no longer active in this connection.

No work of any consequence has been done by Lurgi on the processing of high- or low-temperature tars by chemical or solvent extraction methods.

The following circulars and reprints of articles were furnished by Dr. Oetken:

Recent Developments of Low-Temperature Carbonization of Solid Fuels in Germany, Dr. F. A. Oetken, May 27, 1936, reprinted from Engineering Progress, October 1936.

Lurgi Carbonizers.

Krupp-Lurgi Kammer-Schwelanlagen für Steinkohle.

Gegenwärtiger Stand der Entphenolung von Abwässern aus Kohleveredlungsanlagen.

Die Druckvergasung Fester Brennstoffe mit Sauerstoff von Dr.-Ing. Friedrich Danulat, Frankfurt am Main.

Edeleanu Gesellschaft m.b.H., Berlin

On November 4, 1945, Dr. P. Jodeck of Edeleanu Gesellschaft accompanied Dr. W. F. Faragher (Liquid Fuels Group) and E. O. Rhodes to his office on Martin Luther Strasse (61-66) and tried to find drawings covering the design of the suction filter used at Espenhain for the dewaxing of brown-coal tar distillate. Only a few of the desired drawings were found.

The filter mentioned is made by Maschinenfabrik Buckau, formerly R. Wolf A. G., Magdeburg-Buckau, and is called the R. Wolf-Bandzellenfilter. Each filter unit consists of 40 individual filter cells or pans, each about 6 feet long, 17 inches wide, and 5 inches deep, mounted on an endless-chain conveyor and arranged for automatic filling, application of suction, and discharge of

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the cake with compressed air or inert gas. Each cell has a fabric or paper diaphragm as the filter medium, and 17 cells are in use at any one time. The linear speed is about 4.9 feet per minute. The filter is described and illustrated in C. I. O. S. file XXXI-85, item 30 (PB 1,850), "Edeleanu G.m.b.H., Altenburg, Germany", 1945, by J. P. Jones; also available as a part of TOM Microfilm Reel 198. Figure III from the report by Jones is reproduced as figure 26 in this report.

In using the filter, one part of the brown-coal tar distillate is mixed with three-fourths part of the dichloroethene. The mixture is cooled to 5° C. (41° F.) in a double-pipe chiller and then is filtered by the cells. Vacuum is regulated to suit the conditions and type of oil to be filtered but is within the range 0.3 to 0.5 atmosphere or 4.4 to 7.3 pounds per square inch (absolute pressure). When brown-coal tar oils are filtered in this manner, the wax contains a trace to 0.1 percent oil. Wax from mineral oils contains 3 to 4 percent oil. The capacity of the filter at Espenhain is 50 to 70 metric tons of distillate per day.

The filtrate from the filter cells is chilled to minus 24° C. (-11.2° F.) and filtered again to make low pour-point lubricating oil.

The extracting and dewaxing departments of the Espenhain plant were not seriously damaged and can be operated if raw materials are made available, but the tar-distillation plant was rather completely destroyed. The experimental extracting and dewaxing plant at Rositz also was badly damaged, according to Dr. Jodeck.

A copy of an article by Prof. Ernst Terres, entitled "Die Zerlegung von Schwelteeren aus Braunkohle und Ölschiefer mit selektiven Lösungsmitteln," was presented to the writer by Dr. Jodeck. It describes the method of refining used for brown-coal tar that was developed at Rositz and employed at Espenhain. A translation of this article is included as Appendix A in the above-mentioned report by J. P. Jones.

Fried. Krupp Aktiengesellschaft, Essen, and Krupp Treibstoffwerke G.m.b.H., Wanne-Eickel

Introduction

Two meetings were held with members of the Krupp organization; the first, on September 14, with Dr. F. Müller, director, and Dr. W. Demann, chief chemist, and the second, on September 20, with Dr. Demann, Dr. Junkermann (Engineer in charge of development of low-temperature carbonization plant at Wanne-Eickel), and Mr. Combles, director of the plant at Wanne-Eickel.

The first meeting was held in Director Müller's office in the main office building of Krupp in Essen. The second meeting was held in Director Combles' office at Wanne-Eickel. Following the latter meeting, a brief inspection of the Krupp-Lurgi low-temperature carbonization plant was made.

The principal purpose of each of these visits was to obtain information concerning the production and use in Germany of low-temperature tar in general and low-temperature tar of the Krupp-Lurgi variety in particular.

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Production of Low-Temperature Tar in Germany

Director Müller confirmed the information previously furnished by Dr. Oetken of Lurgi concerning the locations of the low-temperature carbonization plants in Germany. The only plants actually operating on bituminous coal during the war were either of the Krupp-Lurgi or the Lurgi-Spülgas type. The Krupp-Lurgi plants were situated at Wanne-Eickel (Krupp Treibstoffwerk) and Velsen (Saargruben A. G.). The original experimental plant of this type was built by Krupp at its Amalie plant near Essen. Another experimental plant of the same type was installed by Saargruben A. G. at its Heinitz plant near Neunkirchen (Saar). The Lurgi-Spülgas plants were at Blechhammer, Oswiecim (Auschwitz), and Beuthen. A Brennstoff Technik plant (designed by Puening) was under construction but was not completed. Also, a Koppers low-temperature or, more correctly, mid-temperature plant was under construction at the Oheim mine in Upper Silesia but was not completed.

The Krupp-Lurgi Process

Dir. Müller commented on the fact that actual use of the Krupp-Lurgi type of plant for several years, first on pilot-plant scale and then on a large commercial scale, had thoroughly demonstrated its suitability for the low-temperature carbonization of caking bituminous coals. He believes that it is the most suitable type of equipment yet developed for this purpose.

Concerning the products made by the Krupp-Lurgi process, Director Müller stated that the coke has been found to be especially suitable for making water gas for the Fischer-Tropsch process or for the recovery of hydrogen, because the hydrogen content of the water gas is somewhat higher than that of water gas made from high-temperature coke. Because of the better ratio of H_2 to CO in the water gas from Krupp-Lurgi low-temperature coke, the water gas requires less catalytic conversion (or, rather, a smaller proportion of the gas needs to be converted) to make synthesis gas for the Fischer-Tropsch process. Director Müller also said that the Krupp-Lurgi coke does not clinker in the water-gas generator, and therefore ash removal is easier, and less unburned carbon remains in the ash. The highest temperature reached in the generator when using Krupp-Lurgi coke is 50°-100° C. (122°-212° F.) lower than the temperature in the same zone when high-temperature coke is employed. This is due to the greater reactivity of the low-temperature coke. This property was also mentioned in connection with the suitability of the Krupp-Lurgi coke for domestic heating purposes. Its uniform size, freedom from dust, low ignition temperature, and freedom from smoke also were mentioned in this same connection.

Use of Low-Temperature Tar, Particularly for Fuel Purposes

Most of the low-temperature tar made from bituminous coal during the war was hydrogenated or used by the German Navy for fuel. A small amount was furnished to Gesellschaft für Teerverwertung. In an interview with that company, it was learned that the low-temperature tar was mixed with fuel oils derived from high-temperature tar.

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In connection with the use of Krupp-Lurgi low-temperature tars as fuel, Dr. Demann said that some of the advantages of Krupp-Lurgi tar over other low-temperature tars are higher specific gravity (above 1.0), which facilitates the separation of water; a lower "stockpunkt," which indicates greater fluidity at low temperature; and somewhat greater miscibility with oils that are not entirely aromatic.

The subject of miscibility has received a great deal of attention, according to Dr. Demann, because of the need for mixing various fuel oils used by the Navy. The studies showed that all aromatic oils may be blended in any proportions, but sludging takes place when aromatic and paraffinic oils are mixed. It was found that the low-temperature tar could be mixed with aromatic fuel oils from the Fischer-Tropsch synthetic fuel plants or from the Bergius hydrogenation plants. It also was found that the addition of phenols or phenylhydrazine to the low-temperature tar would prevent sludging when mixtures containing paraffinic oils were prepared. However, to avoid the use of such admixtures, attempts were made to remove the asphaltenes from the tar before adding it to the paraffinic oil. The procedure developed for this purpose consisted in diluting the low-temperature tar with benzene in the proportion of 10 parts benzene to 1 part tar. The asphaltenes that are precipitated by the benzene are separated from the liquid portion of the benzene-tar mixture by decantation, and the latter is then extracted with dilute caustic soda to remove phenols and distilled with enclosed steam to remove the benzene. The extraction with caustic soda and distillation with benzene, according to Dr. Demann, must be done at low temperatures to avoid the formation of more asphaltenes. Vacuum is used during the distillation process. The asphalt and tar acid-free oil made in this manner may be used to make additional low-temperature tar usable with paraffinic oils. Separation into two layers is not entirely prevented, but Dr. Demann says that this is not objectionable so long as precipitates are not formed. The yield of asphalt-free low-temperature tar extract made in the manner described above was said to be 80 percent, the balance being asphalt and phenols.

The economic situation with respect to the disposal of low-temperature tar was discussed at some length. Dr. Demann agreed that although it has been profitable to carbonize bituminous coal by low-temperature methods with assured outlets and good prices for the coke (to Fischer-Tropsch), tar (to hydrogenation or Navy fuel), and light oil (to motor fuels), it will be necessary to find new uses at good prices for the tar if low-temperature carbonization is to be profitable in Germany in the future. It is recognized that products from low-temperature tar are not interchangeable with those from high-temperature tar, and special manufacturing procedures or uses must be developed.

In his investigations of the miscibilities of aromatic and paraffinic oils, Dr. Demann found that the Höppler viscosimeter can be used to advantage. Mixtures of the two materials under investigation are made in different proportions, and the viscosity of each mixture is determined with two balls of different size. Mixtures which show no tendency to sludge give the same viscosity reading with either ball, but the two determinations are different when there is actual or incipient sludging. By this means, ranges of

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miscibility and nonmiscibility may be established for mixtures of aromatic and nonaromatic oils. This may be of interest to users of creosote-petroleum mixtures in the United States.

As reference material on the various subjects discussed above, Dr. Demann supplied copies of the following papers:

Wassergaserzeugung aus Steinkohlen-Schwelkoks, Dipl. Ing. Wilke "Technische Mitteilungen Krupp," April 1937, pp. 44-49.

Kohlenschwelung (pp. 177-183 on Steinkohlenschwelteer) by Adolf Thau, Verlag von Wilhelm Knapp, Halle (Saale), 1939.

Über Betriebserfahrungen mit der Steinkohlenschwelanlage Bauart "Krupp-Lurgi" auf Schachtanlage Amalie der Fried. Krupp Aktiengesellschaft, Bergwerke, Essen, von K. Brüggemann; Technische Mitteilungen Krupp, July 1938, pp. 50-58.

Über die Schwelung der Steinkohle in Verbindung mit der Fischer-Tropsch-Ruhrchemie-Synthese, F. Müller, Technische Mitteilungen Krupp, July 1938, pp. 47-49.

Die bisherigen Arbeiten der Vereinigung für Steinkohlenschwelung (V.f.S.), Dir. H. Weitenhiller, Glückauf, No. 35, 1939.

Laboratoriumsapparatur für Steinkohlenschwelung nach dem Heizflächen-verfahren, K. Schoeban, Forschungsberichte, Jan. 1940, pp. 39-44.

Mischbarkeit von Heizölen, Dr. W. Demann, Glückauf, No. 5, 1940, pp. 61-68.

Die Bestimmung der Pumpfähigkeit von Heizölen, H. R. Asbach and J. Tagethoff, Forschungsberichte, Feb. 1943, pp. 52-56.

Viskosimetrisches Verfahren zur Bestimmung der Mischbarkeit von Kohlen-wasserstoffgemischen, insbesondere von Heizölen, W. Demann and H. R. Asbach, Forschungsberichte, Feb. 1943, pp. 42-51.

Die physikalische Prüfung von Schwel-und Hochtemperaturkoks in einer 5-kg-Trommel, E. Combles and W. Surmund, Forschungsberichte, Feb. 1943, pp. 38-41.

Fischer-Tropsch and Krupp-Lurgi Plants

Following the discussions outlined above, the operations of the Fischer-Tropsch plant and of the Krupp-Lurgi plant were discussed briefly with Dr. Demann, Dr. Junkerman, and Director Combles. The location of these plants at Wanne-Eickel are shown by a "Lageplan," and the more essential information concerning each is given on two flow diagrams dated May 25, 1945, which were supplied by Dr. Demann.

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No detailed inspection was made of the Fischer-Tropsch plant. Furthermore, inspection of the low-temperature plant was superficial because this target has been covered by previous investigators. However, a few points of special interest were noted.

Two of six water-gas generators had been operated on low-temperature coke and the remainder on high-temperature coke. A new method of charging the ovens has been developed, which was claimed by Dr. Junkermann to be superior to the previous method. Instead of feeding the fine coal into the coking chambers in small amounts, with tamping between increments, the coal is dropped by a special mechanism into all of the chambers simultaneously with enough force to obtain greater density than formerly in the finished coke.

The bottom closure has been improved. Instead of the two hinged doors previously used, each chamber is now provided with a grating, which is moved into position to close the bottoms of the chambers while a door that covers the entire bottom of the oven is lowered by means of a hydraulic cylinder and moved from under the oven by the car on which the hydraulic cylinder is mounted. The coke is discharged when the grating is moved to the open position. When the door is returned to the bottom of the oven, it is made gas tight by a water seal.

The metal of which the ovens are constructed throughout is ordinary boiler plate steel. Director Combles referred to a book that called it "open-hearth steel" or "plain carbon steel of the "beruhigter Stahl' (killed-steel) type." He said that cheaper steel had failed by blistering and that the use of more costly steel is unwarranted. Ovens built of boiler steel are in good condition after several years of use and are believed to be good for at least 10 years.

The metal conveyor along the bottom of the coke wharf is of a vibrating type, which was said to be superior for the purpose to belt or other types of conveyors. It is called "Schwingrinne" and is made by Schenk in Darmstadt.

In the coke-screening and sizing building a special machine breaks the coke slabs into small pieces with very little formation of fines. The slabs roll, by gravity, over spiked rollers, between which are openings. Pieces that will not go through the openings between the rollers are broken by spiked bars attached to wooden planks, which are pulled upward by friction between cams and dropped when the friction is released. Other precautions are taken to avoid degradation, such as the use of a conveyor for loading cars, which can be lowered to the bottom of the car to be filled, thus avoiding the dropping of the coke from any appreciable height.

Except for damage to some pipe connections and to the buildings and one or two ovens, the low-temperature plant appeared to be in fairly good condition. It was explained that repairs had been made after the earlier raids, and no great amount of damage was suffered from the last raid. On the basis of cost, the plant was said to have been damaged to the extent of 33 percent, the total cost of the plant having been 6,000,000 marks and the total estimated cost of the damage 2,000,000 marks.

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In the case of the Fischer-Tropsch plant, the total cost of the plant was said to have been 40,000,000 marks and the total damage 10,000,000 marks.

It was stated by Director Comblés and Dr. Junkermann that both plants could be placed in operation shortly after the first of the year if permission is granted to resume operations.

Braunschweigische Kohlen Bergwerke, Helmstedt, Schwelwerk Offleben

Schwelwerk Offleben was visited on November 5 with Major E. E. J. Farmer, district representative of the North German Coal Control, and Herr Schmidt, of Braunschweigische Kohlen Bergwerke (B. K. B.). Herr Schmidt acts as liaison officer between the Military Government and B. K. B. and also as interpreter. Ing. Teuscher, assistant superintendent, helped explain the operations of the plant.

Schwelwerk Offleben is one of three installations which together comprise B. K. B.; a second installation is in the British Zone, and the third is in the Russian zone. There is only one coal-cleaning plant to serve the three groups. It is in the British zone. On the other hand, power is furnished to the three installations from a power plant in the Russian zone.

The Offleben brown-coal mine was visited first. It uses modern digging equipment for removal of overburden and brown coal and for backfilling the excavation. The normal output of the three mines is about 14,000 metric tons of brown coal per day. Total capacity is about 20,000 metric tons.

Most of the brown coal from the three mines is used either in loose or briquetted form for fuel, but a part of the Offleben production goes to the Lurgi-Spülgas plant. In this plant the raw coal goes first to a bunker, from which it is conveyed to crushers, which reduce it to 0-20 mm. (0-0.7874 inch) size. It is then dried to about 15 percent moisture content by recirculated gas and is briquetted by pressure only. The briquets are loaded into the top sections of the carbonizing retorts, where the remainder of the moisture is driven off by combustion gases. The briquets disintegrate on drying, and the granular semicoke passes downward in the presence of distillation gases (temperature, 600°-700° C. or 1,112°-1,292° F.). The granular coke passes through a cooling zone and is removed from the oven through a discharging device.

Tar and oils are removed from the gas by electrical precipitators, indirect coolers, and Feld washers. The tar is shipped to hydrogenation plants. The middle oil is used as the scrubbing medium for the light oil but eventually is hydrogenated or worked up for gasoline and Diesel oil. The benzine fraction also is further refined for use as motor fuel. The tar, oil, and benzine are worked up by Braunkohle-Benzin A. G. The capacity of the retorts is about 3,300 metric tons per 24 hours. When visited, it was running at about one-third capacity. A description of the process follows.

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Description of Offleben Low-Temperature Distillation Works

Raw brown coal with about 54 percent moisture is transported from open-cut mines by electric railway to the carbonization plant and unloaded into a low bunker of 5,000 metric tons capacity. This bunker insures a continuous supply of coal for the plant in case of interruptions in coal production and in deliveries on Sundays and holidays. The coal is lifted from the bunker by bucket excavators and dumped onto a belt, which conveys it to the so-called "Nassdienst" plant, where the raw coal is prepared for the drying process. Here the coal first passes a roller grate and is screened. The coarse material is crushed by "Titan" breakers and, together with the fine coal (0-20 mm. or 0-0.7874 inch size), is transported to the "Trockendienst" or drying plant, which reduces the moisture to a suitable percentage for briquetting.

The Offleben distillation works is subdivided into a briquetting plant, a gas-producer plant, and a low-temperature carbonization plant. The finely crushed raw coal is dried in inert-gas driers and briquetted with plunger presses without use of binder. For drying, surplus gas from the carbonizers is used principally, together with a small quantity of producer gas. Briquetting the dried coal prepares it in suitable form for efficient carbonization by the Lurgi-Spülgas process using direct heating with high-temperature recirculated gas, and, in addition, makes possible the production of coarse granular coke.

The briquets manufactured in the briquetting plant are conveyed by rubber belts into the carbonizing plant and charged into 10 Lurgi retort-type ovens. The Lurgi oven consists of two main parts: The upper part, or predrier, and the lower part, or carbonizer. The two parts are joined by eight connecting channels. Each oven is constructed as a vertical double oven, between which are combustion chambers and gas channels. The combustion chambers are made of fire brick, and the predrier and carbonizer are brick-lined. The baffles inside of the predrier are made of steel, and the louvers in the carbonizer are of ceramic material. Exclusive of the coal-charging and coke-discharging arrangements, the height of the retort is about 17 meters (55.8 feet). Any dust accumulated inside the oven can be removed during the operation.

The briquets in the predrier are heated by recirculating gases and dried from about 15 to 0 percent moisture content. They are slowly heated while moving downward by gravity and pass through a maximum drying-gas temperature zone of 220°-300° C. (428°-572° F.), depending on the throughput of the oven.

The dried briquets in the carbonizer are heated by recirculating flushing gas (Spülgas) to a temperature between 600° and 700° C. (1,112° and 1,292° F.). The heat required comes from carbonization gas burned in a combustion chamber. After passing through the carbonizing zone, the char is cooled down by recirculated carbonization gas to a temperature of about 120°-200° C. (248°-392° F.), depending on the throughput of the oven.

The carbonization gases leave the carbonizing zone, are collected in the upper part of the carbonizer, and leave the oven at a temperature of about 240° C. (464° F.). They are first cleaned from dust, are then cooled in a

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direct water-spray cooler, where some heavy tar is taken out, and are next passed through an electrical tar precipitator, which removes most of the remaining tar. The gas is further cooled in indirect coolers, and middle oil and water are condensed out. The middle oil, after removal of benzine, is used as wash oil to recover light oil (crude benzine) from the carbonization gas. This washing takes place in vessels of the Feld type, in which the precleaned gas comes in direct contact with wash oil in a very fine spray. The light oil washed from the gas is recovered in a benzine-distillation plant operated with steam. A part of the used benzine-free wash oil leaves the system and, together with the heavy and light tar, is shipped to hydrogenation plants for conversion into motor fuels. A part of the crude benzine is refined, but most of it is also hydrogenated.

During the drying and carbonization processes, the briquets are converted into coke containing both coarse and fine materials. The coke, after discharge from the oven, is first cooled and stabilized in four revolving drums made of sheet iron. The coke in the drums is sprayed lightly with water and treated with an air-steam mixture. The cool coke is conveyed to a bunker of 2,500 metric tons capacity, where the coke is screened into three different sizes, namely, fine coke from 0-10 mm. (0-0.4 inch), middle size from 10-20 mm. (0.4-0.8 inch), and coarse coke above 20 mm. (0.8 inch). The fine size is used in boiler plants and in Winkler gas producers; the middle size is shipped principally to central-heating and gas-producer plants; whereas the coarse coke finds its main market as household fuel. The dry coke has a calorific value of about 6,600 kg.-cal. per kilogram (11,800 B.t.u. per pound) and contains about 18 percent ash. After direct sprinkling with water in the drums, the coke contains up to 18 percent moisture and has a calorific value of about 5,300 kg.-cal. per kilogram (9,540 B.t.u. per pound).

The carbonization gas, which has a calorific value of about 2,200 kg.-cal. per cubic meter (247 B.t.u. per cubic foot), is used for heating the predrier and the carbonizer in the carbonization plant. Surplus carbonization gas is used for drying the raw brown coal in the drying system ahead of the briquetting plant. A small quantity of the carbonization gas is sent to the boiler plant at the "Treue" mine to generate steam.

Each Lurgi oven has a capacity of 300 to 400 metric tons of briquets per day, which for nine operating ovens amounts to about 3,000 to 3,300 metric tons per day, or about 1,200,000 metric tons per year - the equivalent of 2,100,000 metric tons of raw brown coal. The tar content of the briquets is between 10 and 12 percent. Yearly production of tar, middle oil, and crude benzine amounts to about 84,000, 18,000, and 18,000 metric tons, respectively. In addition, 600,000 metric tons per year of coke in three different sizes is produced. The tar, middle oil, and crude benzine are shipped to Braunkohle-Benzin A. G. for hydrogenation. After the war, the tar was used in coke-oven plants of the Reichwerke in Watenstedt for the production of gas.

Storage facilities at the Offleben works include six tanks with a total capacity of 7,000 cubic meters (1,849,210 U. S. gallons) for storage of tar and oil, one 500 cubic-meter (132,090-gallon) tank for middle oil, and one 500 cubic-meter (132,090-gallon) tank for crude benzine. All products are shipped from the works by railroad.

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The electrical energy for the Offleben works is supplied by the Harpke power plant. The yearly power demand at full capacity amounts to 55,000,000 to 60,000,000 kilowatt hours. About 800 cubic meters (211,340 U. S. gallons) per hour of recirculating water, which is cooled in specially designed towers, is required for cooling purposes.

In addition to the plants mentioned above, the Offleben works has the usual auxiliaries found in modern works that are required for efficient operation and for the welfare of plant personnel.

I. G. Farbenindustrie, Merseburg-Leuna

The Leuna plant of I. G. Farbenindustrie near Merseburg was visited during the week of December 30, 1945. The following processes were inspected:

1. The hydrogenation of brown coal.

2. Continuous distillation of the product from liquid-phase hydrogenation of brown coal to produce an oil distilling at 325° C. (617° F.) and a residue for reuse as pasting oil.

3. Continuous extraction of phenols from the distillate boiling below 325° C. (617° F.) recovered by the continuous distillation mentioned in (2) above.

4. Continuous distillation of the phenols to obtain forerunnings, a fraction high in phenols, a fraction low in phenols, and a residue.

5. Springing and recausticizing of the phenolate and carbonate from the continuous extraction plant.

6. Refining of phenol and cresols recovered in the above processes and received from other plants (Most, Pölitz, and Blechhammer).

7. Manufacture and distillation of cyclohexanol by the catalytic hydrogenation of phenol, using a nickel-aluminum oxide catalyst.

8. Manufacture of cyclohexanone by the catalytic dehydrogenation of cyclohexanol, using a zinc-iron catalyst.

9. Manufacture of oxime from cyclohexanone, using a hydroxylamine sulfate solution made from ammonium bisulfite, ammonium nitrite, and sulfur dioxide.

10. Production of caprolactam or luran by reacting the oxime with sulfuric acid followed by neutralization with a solution of ammonium hydroxide.

Descriptions of these processes, with flow diagrams, were obtained; also, descriptions and flow diagrams for small-scale plants that were used for making brenzcatechin (pyrocatechol) and adipic acid. These two latter

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plants had been destroyed by air raids and therefore could not be inspected. The other plants listed above had been damaged, but each could be used, at least at part capacity, except the continuous extraction plant.

A plant for the extraction of phenols from waste waters by means of tricresylphosphate was inspected. It was heavily damaged by bombs. A flow diagram (without description) for this plant was received.

A pilot plant that had been used for trial of the phenosolvan process had been completely destroyed and could not be inspected. No description of this plant was available.

During the period of January to May 1944 the Leuna plant processed 2,500 metric tons of tar acids per month. It was received from the following sources:

	Metric tons
Produced at Leuna by the hydrogenation of brown coal	870
Phenosolvan extract from Politz hydrogenation plant	320
Phenosolvan extract from Most (Brüx) hydrogenation plant	1,150
Phenosolvan extract from Blechhammer hydrogenation plant	160
	2,500

The following monthly quantities of products were made:

	Metric tons
Crude phenol	15
Pure phenol	265
Ortho fraction	80
Ortho cresol 50 percent	90
Cresol DAB_4 (at least 38 percent m-cresol)	360
Cresol DAB_6 (at least 50 percent m-cresol)	40
Xylenol	230
Xylenol N	70
Crude Brenzcatechin I	25
Crude Brenzcatechin II	130
Crude Brenzcatechin III	2
Crude Brenzcatechin IV	70
Residues returned to hydrogenation	710
	2,087

Braunkohle-Benzin A. G. (Brabag), Berlin

The investigator went to Schinkelplatz on November 1, 1945, but found the office building previously occupied by Braunkohle-Benzin A. G. to have been almost completely destroyed. A sign on the door read "Die Verbindungstelle, Karlstr. 20A N. W. 7." Because of lack of time, this address was not visited.

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From other sources the writer was informed that Brabag has four plants in operation, which since the war were first under Russian supervision and later were declared possessions of the Russian Government. Three of these plants are hydrogenation plants and are at Böhlen near Leipzig, at Tröglitz near Zeitz, and at Rothensee near Magdeburg, whereas the fourth is a synthesis (Fischer) plant near Schwarzheide. The hydrogenation plant at Rothensee, meanwhile, has been completely dismantled and transported to the East. Forty Fischer ovens of the Schwarzheide plant have been given to the Poles, and it is said that they will start a new synthesis plant in Upper Silesia.

Brennstoff Technik, Essen

On October 17, 1945, Director Edw. Kuhl was interviewed in Essen concerning the work done by Brennstoff Technik toward the development of a process invented by Franz Puening for the low-temperature carbonization of coal. Brennstoff Technik was organized to promote the use of this process. The company was capitalized at 1,200,000 R. M. At the time of the interview, it had spent between 1,000,000 and 1,200,000 R. M. on development work.

An experimental plant was erected at the Gas Works at Berlin which had a capacity on coking coal of 50 metric tons per 24 hours and a capacity of 100 metric tons per 24 hours when operated on noncoking coal or coal briquets.

A large plant of 16 ovens with a daily capacity of 1,600 metric tons of coal briquets was under construction at the Hermann Goering Works near Saarbrücken and was about 50 percent completed when the war ended. Another plant of the same capacity had just been started at the Hermann Goering Works near Katowice (Kattowitz).

Experimental work on the tar from this process was conducted by Chemische Fabrik, Byk-Gulden Werke, of Berlin. A pilot plant of 5 to 6 metric tons daily capacity was operated to make fuel oil, Diesel oil, and phenolates. Two large continuous tar plants were projected (but not started) for the two Hermann Goering plants mentioned above.

At the conclusion of the interview, Director Kuhl offered to prepare a report giving more detailed information about the B. T. process. The report was received from him on November 7, 1945.

Dr. Lameck, Essen

The following statement concerning the processing of low-temperature tars according to a procedure worked out by Byk-Guldenwerke Chemische Fabrik A. G., Berlin, was furnished by Dr. Lameck of Essen, who at the time of the interview was associated with the Mines Supply Division of North German Coal Control.

> The treatment of low-temperature tars according to the process of Byk-Guldenwerke was developed in the research laboratory at Oranienburg by the chief chemist, Dr. Hansen, as far as it concerns the mode of procedure. The technicalities were developed by the firm of Brennstoff Technik G.m.b.H., Essen, Bürohaus Städtische Sparkasse.

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An experimental plant with a throughput of about six metric tons of tar per day was arranged at Oranienburg in 1942 by orders of competent authorities of the Reich. The plant was destroyed by bombs at the beginning of this year.

Up to 1945 many experiments had been made with various tars yielded by carbonization of long-flame coals from the mining districts of Upper Silesia, the Saar, and Lorraine. On account of the satisfactory tests, Byk-Guldenwerke was able to offer a process for practical use. Together with competent authorities of the Reich two works projects were finally settled. These projects concerned the design of plants in Upper Silesia and Lorraine.

(1) Treatment by Distillation

When distilling a true low-temperature tar the yield of distillate amounts to about 65 to 70 percent and that of pitch to 35 to 30 percent. The lower and upper boiling limits of the distillate are between 180° and 360° C. (356° and 680° F.). Small quantities of benzine (octane rating about 80) may be left out of account. The pitch may be used as a binding substance for the briquetting of coal in the customary way. Also, it is a raw material for hydrogenation or for the manufacture of electrode coke. The distillate is treated by sodium hydrate to extract the phenols. These phenols, including their high boiling ingredients, were used by Byk-Guldenwerke for the manufacture of synthetic plastics and lacquers. The resulting neutral oils had been found suitable for use in slow-running Diesel engines. By treatment with methyl alcohol it is possible to divide this oil into a portion easily ignitable (octane rating about 39) and into another portion of a high ignitability (octane rating about 20). The properties of Diesel oils are naturally dependent on the qualities of the tars to be treated. The qualities are dependent on the conditions of carbonization, i.e., rate of temperature and process of carbonization. It has been proved that the best tars were obtained by heating-surface ovens.

(2) Process of Washing

As there is no difference between the specific gravities of tar and sodium hydrate, it is necessary to dilute the tar at first. Then it is possible to extract the phenols by sodium hydrate. After distilling the dilution agent, the remaining neutral oil may be used as qualified heating oil or in slow-running Diesel engines. Otherwise, the neutral oil may be divided into pitch and Diesel oil (octane rating about 28) by distillation. As mentioned, it is further possible to divide this oil into a portion easily ignitable and another portion of a high ignitability by treatment with methyl alcohol.

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Heinrich Koppers, G.m.b.H., Essen

The firm of Heinrich Koppers G.m.b.H., Essen, was interviewed in November 1945 concerning the low-temperature carbonization of coal.

In 1942, the Koppers organization received an order from the Bergwerksverwaltung Oberschlesien in Kattowitz for a low-temperature carbonization plant with a capacity of 1,000,000 metric tons of noncaking and weakly caking bituminous coals per year to be installed at the Oheim mine. The design of the installation was based upon past experience in the use of continuous, vertical, refractory ovens for high- and medium-temperature carbonization purposes. Also, this experience was supplemented by large-scale tests conducted at the Oheim mine of Bergwerksverwaltung Oberschlesien. The plant was not completed before the end of the war.

The Koppers low-temperature plant was to have vertical retorts each 11,500 mm. high, 3,500 mm. long, 250 mm. wide at the top, and 350 mm. wide at the bottom. Equivalent English units are 37.7 feet, 11.5 feet, 9.8 inches, and 13.8 inches, respectively. The following yields were expected per metric ton of coal carbonized: Coke (3 percent moisture), 64.0 percent; gas, 300.0 Nm^3 (11,390 cubic feet at 60° F. and 30 inches Hg pressure, saturated); low-temperature tar, 9.3 percent; and low-temperature benzine, 0.8 percent.

Tar produced in the test plant at the Oheim mine was distilled continuously with a pipe still to determine the yields of products that might be expected. Heating oil, 70 percent, pitch (82° C. or 179.6° F.), 29 percent, and light oil, 1.0 percent, were produced.

Additional details concerning this project are furnished in a report entitled "Die Schwelung der Steinkohle nach Koppers."

Firma Carl Still, Recklinghausen

On November 9, 1945, Mr. Still and Dr. Schmidt of the Carl Still organization, 21 Kaiserwall, Recklinghausen, were questioned concerning equipment furnished for the low-temperature carbonization of coal or for the processing of low-temperature tar. They reported that in 1942 their organization had built a plant at Espenhain near Leipzig for the processing of 200,000 metric tons of brown-coal tar per year. The plant was built for Aktiengesellschaft Sächsische Werke. It consisted of a centrifuge plant for removing suspended matter from the brown-coal tar; a continuous pipe still operated atmospherically; a vacuum pipe still installation to further distill the residue from the atmospheric unit; batch stills for blowing the soft pitch from the vacuum still with hot inert gas to make hard pitch; and a pitch coke plant.

For processing the distillates, there was an Edeleanu extraction plant to remove paraffins and batch redistilling stills with columns.

Descriptions of most of these units were furnished by Mr. Still at a later date.

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Gesellschaft für Teerverwertung, Duisburg-Meiderich

Following a report that 28,000 metric tons of low-temperature tar from bituminous coal had been furnished to this firm for distillation purposes in 1943, members of the firm were interrogated on September 17, 1945.

It was stated that low-temperature tar from bituminous coal carbonized in Krupp-Lurgi retorts had been received from Wanne-Eickel at the rate of 400 metric tons per month and from the Saar at the rate of 300 metric tons per month. However, instead of being distilled at Meiderich, the low-temperature tar was mixed with other materials distributed for fuel purposes from that plant. The proportion of low-temperature tar in the fuel mixture was 10 percent.

In July 1944, a small quantity of low-temperature tar produced by Brennstoff Technik was furnished to Gesellschaft für Teerverwertung for test purposes. A copy of a report covering a batch distillation of this tar was received, as well as a copy of an article by Dr. O. Kruber entitled "Beiträge zur Kenntnis des Steinkohlenschwelteeröls."

Julius Pintsch, Kommanditgesellschaft, Berlin

The office of Julius Pintsch, Andreas Str. 71-73, Berlin, was visited for the first time on November 2, 1945. The person interviewed was Director Dr. H. Rosenthal. For information about the Pintsch-Hillebrand process, he suggested an interview with Dr. Domann on the following day. For information about tar processing, he suggested that Dr. Stock be interviewed when available.

Dr. Stock was contacted the next morning at his home, Nelkerstr. 3, Berlin-Zehlendorf. He said that Pintsch builds plants for refining the benzene recovered at brown-coal distillation plants and from the distillation of brown-coal tar. The benzine, in vapor phase, at 300°-400° C. (572°-752° F.) is passed over iron ore. Iron sulfide is formed and the liberated hydrogen acts as a hydrogenating agent, and subsequent refining losses are decreased. No corrosive sulfur is left in the benzine. According to Dr. Stock, the process can be used on brown-coal tar and Esthonia shale tar. A refining plant of this type was started about 1940. A larger plant of the same kind for refining benzol was operated by Grossgaserei Magdeburg. The process was patented in Germany by Dr. Rostin. The patent is owned by Continental-Öl, but Pintsch has the right to build plants in Germany. The process was described by Director Jehn in "Die Braunkohle" during the period 1940-42. Dr. Stock said that he would try to find a copy of the article and send it to the writer. He would like to see the process applied to the shale oil produced at Messel, near Darmstedt, and also to the lignite tar of Hefrag (Hessen-Frankfurt A. G.) in Wölfersheim.

On November 3, the Pintsch oil gas plant at Rummelsburg, in the outskirts of Berlin, was visited with Director Rosenthal. This is one of 26 plants gasifying crude brown-coal tar for the production of gas used by the German railroads for the illumination of passenger cars and also for the

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heating of car wheels in repair shops. Gasification of 1 metric ton of tar requires 200 kilograms (441 pounds) of tar for heating and makes 500 cubic meters (17,660 cubic feet) of gas (calorific value, 9,000 kg.-cal. per cubic meter or 1,010 B.t.u. per cubic foot) and 30 kilograms (66 pounds) of light hydrocarbons (shipped to Rütgerswerke for refining). The gas is compressed to 15 atmospheres (220 pounds per square inch) for distribution in pipe lines to points of use. The plant at Rummelsburg has three machines. Each consists of a heat exchanger or foreheater for the two checker-filler generators that are alternately heated with tar and carburetted with tar. The temperature in the generators is 800°-850° C. (1,472°-1,562° F.) and the pressure 500 mm. (19.7 inches). The gas production of each machine is 250-500 cubic meters (8,830-17,660 cubic feet) per hour, depending upon the quality of the tar. Pintsch has built plants for distilling brown-coal tar in Czechoslovakia, using the Sadewasser process, but they were not very successful. A batch-distilling plant for bituminous coal tar is being erected at Chemnitz for Stetor (manufacturer of roofing felt). A two-ton still with agitator to operate at 70 mm. (2.76 inches) mercury pressure will be installed. It will operate on tar from gas plants or coke plants.

Following the interview, documents were received concerning the Pintsch-Hillebrand water-gas process and synthesis gas from peat.

Dr. Reerink, Esso

Dr. Reerink, formerly Director of Research of Bergbau Verein, and at the time of this visit, October 8, 1945, employed by the Mines Supply Division of North German Coal Control, was contacted to obtain general information about coal tars in Germany.

Dr. Reerink stated that coal-tar research in Germany had been carried on by Institut für Mineralöl Forschung (Prof. Heinze), Berlin; Deutsche Erdöl A. G., Berlin; Edeleanu Ges. (Dr. Terres); Ges. für Kohletechnik, Dortmund; Byk-Gulden Werke, Berlin; and I. G. Farbenindustrie, Leverkusen. Good authorities on these subjects are also Dr. Kraut, I. G. Farbenindustrie, Berlin, and Dr. Gagemann of Ruhrchemie, Sterkrade-Holten.

Dr. Reerink discussed briefly the use of brown-coal tar for hydrogenation purposes; the use of the Edeleanu process (SO_2) for the recovery from brown-coal tar of benzine, Diesel oil, and paraffins; and the combined distillation, extraction, and hydrogenation process tried by Deutsche Erdöl. Later, a 150,000 metric-ton plant was built at Espenhain, according to Dr. Reerink.

Some general remarks were made by Dr. Reerink about the comparative merits of hydrogenation, Fischer-Tropsch, and Pott-Broche processes for making motor fuels and aviation gasoline.

In connection with coke for the aluminum industry, Dr. Reerink said that pitch coke sold for 90 R.M. per ton during the war and coke made from the extract produced by the Pott-Broche process sold for 180 R.M. per ton.

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Dr. C. Otto and Company, G.m.b.H., Dahlhausen

At the office and plant of Dr. C. Otto and Company, Dahlhausen, Germany, visited on October 16, 1945, the following persons were interviewed: Dr. Stuhlmann, Dr. Brensing, Mr. Schoen, and Mr. Boskamp.

The following information was obtained concerning low-temperature carbonization and low-temperature tar: This company has carried on a considerable amount of investigational work on low-temperature carbonization of bituminous coal. A pilot plant was built in which at first it was tried to carbonize the coal by sensible heat alone. This attempt was not successful and a small coke oven with horizontal heating flues was erected and inside the oven were placed, in vertical position, steel members resembling I-beams. When they were in place, the flanges of the I-beams were close to the walls of the oven and the webs were extended crosswise between the walls. The coal was filled into the spaces between the I-beams; heat was transmitted to the coal through the side walls of the oven and also by the steel plates or webs of the I-beams. At the end of the carbonizing period, the low-temperature coke was dropped from the bottom of the oven into a car. The oven was provided with a regenerator, which was directly in line with and on the same level as the coke oven. The coke produced in this oven was of good quality, but the steel-heating members warped badly and made it difficult to discharge the coke, so the steel members were removed and tests were made with heat supplied only through the side walls. A plant designed on this basis was projected but was not built.

The Otto Company has not investigated low-temperature tar. Dr. Stuhlmann mentioned the fact that a large amount of work had been done on the miscibility of low-temperature tar by Krupp under Director Müller and Dr. Demann. The Otto Company also has not built tar plants for the distillation of low-temperature tar.

Deutsche Erdöl A. G. Berlin

On November 1, Director Ritter of Deutsche Erdöl, A. G., was interviewed in his office at 61-66 Martin Luther Str. He stated that the brown-coal distillation operations of Deutsche Erdöl are carried out in two plants at Regis, near Leipzig, and one plant at Rositz. At the latter plant there is a large installation for the distillation of brown-coal tar by the old method used for this purpose. Also, at this plant there is an experimental unit in which was developed the system of dewaxing, distillation, and coking now used commercially at Espenhain.

Director Ritter said that he was not very familiar with the methods used for processing the tar, and the company's four best experts on this subject had died (one only the week before this visit), but he said that he would try to get a description from the Rositz plant.

Following the interview, several documents pertaining to low-temperature were received from Director Ritter.

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Dr. Thau, Didier Werke A. G., Berlin

Dr. Adolf Thau of Didier Werke A. G., Westfälische Str. 90, Berlin-Wilmersdorf, was interviewed on October 1, 1945. After a brief discussion of low-temperature carbonization and the working up of low-temperature tars, including brown-coal tar, Dr. Thau offered to write a special report on these subjects.

According to Dr. Thau, the distillation of brown-coal tar is best described in "Die Chemie der Braunkohle" by Erdamann and Dolch (published by Knapp in Halle, 1927).

The most interesting brown-coal tar-distillation plant is at Rositz at the plant of Deutsche Erdöl A. G. Dr. Thau did not seem to be aware of the fact that a larger plant of the same type had been erected at Espenhain during the war.

Dr. Thau stated that the only systems now in use in Germany for the carbonization of brown coal are the Geissen and the Lurgi-Spülgas processes.

Reference was made to the fact that Dr. Hansen of Byk-Guldenwerke had applied for a patent on a method for recovering tar acids from low-temperature tar. He did not remember the details.

According to Dr. Thau, a commercial, low-temperature plant of the vertical refractory type was erected by Didier Werke during the war.

Following the visit, an article entitled "The State of Low-Temperature Carbonization in Germany Before the Conclusion of the War" and several supporting documents were received from Dr. Thau.

PART III. - INDEX TO FIAT MICROFILM REEL C-98 (TOM MICROFILM REEL 152, PB L 13,093)

Part A

The Low-Temperature Carbonization of Coal and the Processing, Use, and Distribution of Low-Temperature Tar and Products Derived Therefrom. Documents and drawings.

Part B

Processes and Equipment for the Recovery of Phenols From Industrial Waste Waters. Documents and drawings.

Part C

The "Büttner" Drier. Descriptive pamphlets and drawings.

Part D

The "Pintsch-Hillebrand" Water-Gas Generator and the Production of Synthesis Gas from Peat. Documents and drawings.

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Part E

Copy of a Dissertation by Ing. F. A. Croy entitled "Concerning a Process for the Recovery of Pyrocatechol from Brown-Coal Tar, the Use of the Same on a Large Scale, and the Purification of the Technical Product."

FIAT MICROFILM REEL C-98 OR TOM MICROFILM REEL 152, (PB L 13,093)

Documents collected by E. O. Rhodes, Miscellaneous Chemicals Sub-Committee, JIOA, between September 15, 1945, and January 15, 1946.

Filmed by JIOA.

Date: January 26, 1946.

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1. "Recent Developments of Low-Temperature Carbonization of Solid Fuels in Germany" by Dr. F. A. Ostkon. Abstract by A. Thau in Engineering Progress, vol. 17, October 1396, pp. 229-235. 507-513

2. Krupp-Lurgi Kammer-Schwelanlagen für Steinkohle. Krupp-Lurgi chamber for low-temperature carbonization of bituminous coal. 514-524

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